THE USE
OF THE THERMOMAGNETIC BALANCE
IN CALCULATING
MAGNETIC SUSCEPTIBILITY

by

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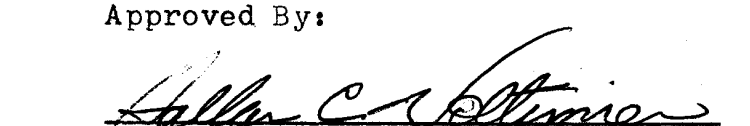
Approved By:



Advisor
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ACKNOWLEDGEMENTS

Thanks is given to Dr. Hallan Noltimier of The Department of Geology and Mineralogy without whose expert instruction this project would not have been possible. Thanks is also given to the Department itself which provided the facilities necessary in the proper completion of this project.

A special thanks to Michael Rhodes who provided the balance itself. The development of the balance and its construction can be found in his Senior Thesis. The work he did was truly extraordinary, for the accuracy of the results shown in the following pages are quite remarkable.

TABLE OF CONTENTS

INTRODUCTION

The following experiment and its results are presented according to each specimen tested and the meaning of its data. Specifically, this consists of calculating the magnetic susceptibility of each specimen, given the field intensity and its field gradient. Eight minerals, all having ferromagnetic properties, were studied. The results of those studies are presented following a basic explanation of the theoretical attempts of this experiment.

THE THERMOMAGNETIC BALANCE

The physical quantity of magnetic susceptibility of a sample is defined as the ratio of the magnetization acquired by the sample in the field to the magnitude of the field. Mathematically it is defined as

$$k = \frac{J}{H}$$

where J represents magnetization strength and H the respective field strength. The value of K is negative and small (10^{-6}) in diamagnetic substances and positive and small in paramagnetic materials. However, in many minerals, the value can be found anywhere from $10\text{-}10^{5}$ in order. These substances are ferromagnetic. The subject of this experiment deals specifically with ferromagnetic minerals (and variations such as antiferromagnetism and ferrimagnetism, which will be sxplained later.)

The magnetic balance, specifically the balance in this experiment, measures the translational force subjected on a sample in an inhomogenous magnetic field (Nagata, p. 46). This translational force is described as

$$F = vJ(H)\frac{\partial H}{\partial y}$$

where $\frac{\partial H}{\partial y}$ is described as the field gradient, v the volume of the material studied, and J(H) the magnetization per unit volume of the sample. Rearranging the equation algebraically to

$$\frac{F}{v\frac{\partial H}{\partial y}} = J(H)$$

one can easily see where the balance can be used to measure J(H)

and hence calculate k. A problem faced by Rhodes was that he had neither measured values for $\frac{\partial H}{\partial y}$ nor H at the time of his studies, but since these have been measured (and can be found at the rear of this paper), k can be calculated.

The translational force F is calculated as the energy of the field times the susceptibility, (which is dependent on temperature, or k_t)

$$Wk_t = \frac{S_2}{S_1}(F_c - F_o)$$

where S_2 represents the lower portion of the pendulum below the pivot and S_1 the upper portion, F_c the force on the control coil (from the ferrite magnet) and F_o the force on the electromagnet. This value of F_o is simply equal to F_c when no sample is in the cup, and can be measured as equal to F_c at the Curie temperature, when

$$F_c = F_o$$

To calculate F_c and in turn calculate F_o, it is necessary to know the moment p about the pendulum magnet. Definitively, the magnet force is defined as H (generated by the coil) times p. However since p is the magnetization of the ferrite magnet, this moment adds to the moment generated by the coil. Therefore F_c at the center of the ferrite magnet is

$$F_c = H_c 2p$$

This moment was calculated by measuring the field at a stipulated distance (6 cms.) from the coil face, a representative distance to the pendulum. When the field had been measured for certain currents (a graph of this data is shown on graph #1), the pendulum was set at this distance x from the coil. Since

$$F \tan a = H_c 2_p$$

and the force F is the force of the pendulum F = mg therefore,

$$mg \tan a = H_c 2_p$$

and since tan a = a for very small angles

$$amg = H_c 2_p$$

The mathematics are shown in the first list of calculations. The moment being calculated, the value of F_c at various temperatures and in turn the value of F_o and Wk_t can be calculated.

Before any samples could be studied, several preparations had to be made. The wooden furnace supports utilized by Rhodes were replaced by permanent aluminum fixtures. The control coil itself was fastened to the base by a thin but sturdy aluminum collar. In addition, an early problem encountered but quickly remedied was the repair of the pendulum itself which was fractured below the cup during the reassembly of the apparatus.

The first measurement taken was the calibration of the balance in the field with no sample and the furnace inoperative. The results of several runs were averaged and tabulated as shown (on graph #2). The purpose of this was to see how accurate the value of the Curie temperature of certain samples were at specific electromagnet voltages. Theoretically, at that temperature, the pendulum balances as with no sample in the cup. To observe any irregularities of the balance being affected by different weights, several runs had been measured with varying masses of aluminum foil in the cup with a sample of magnetite of a known mass. Also masses of aluminum up to 1.0 grams had been used. The results were completely negative, no change had been effected.

The furnace was also calibrated and the results are shown as

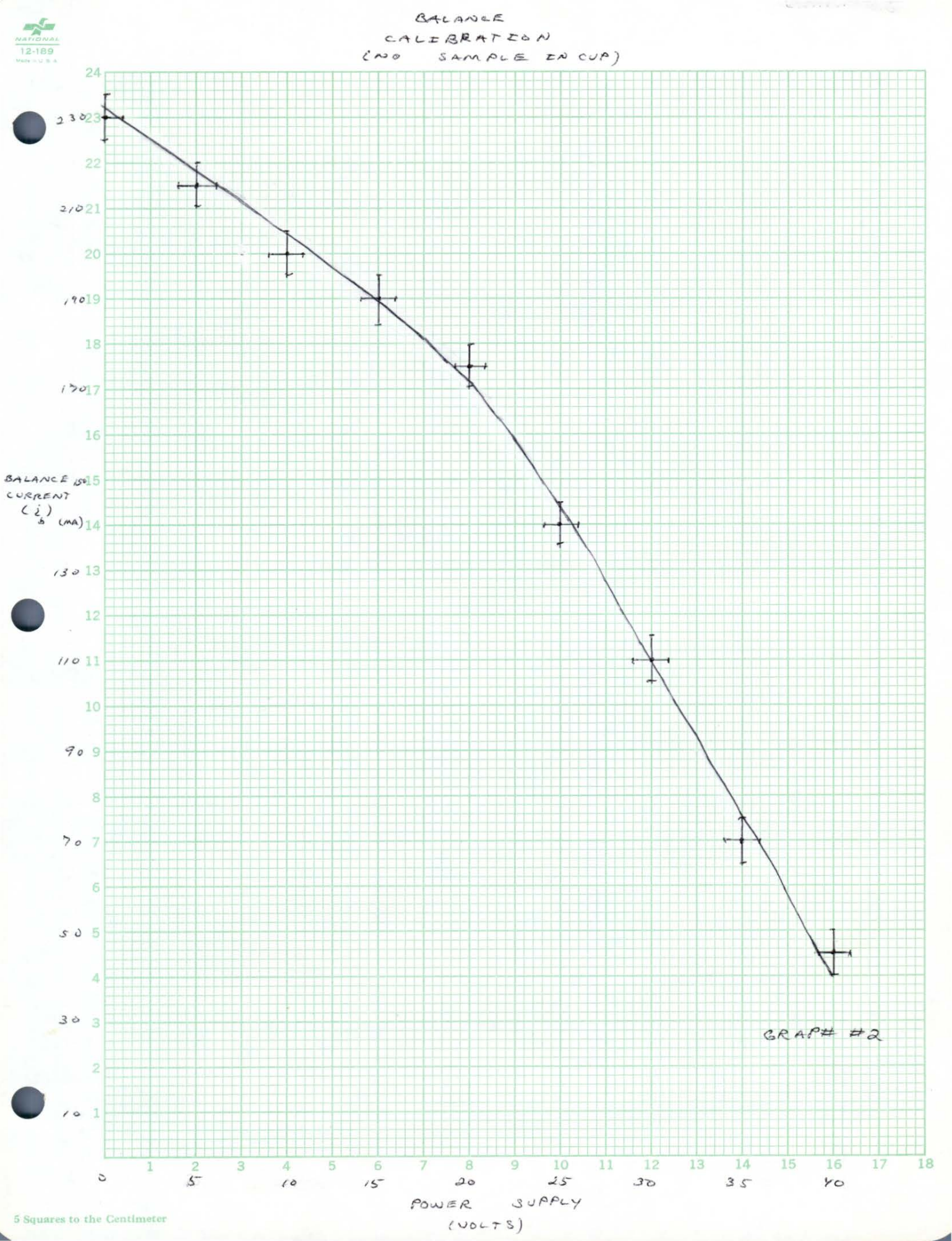
graphed. The linearity of the time versus temperature scale (graph #3) was extremely helpful in getting accurate temperature readings at differing voltages. The two scales are two separate calibrations. The red represents an early attempt with the thermocouple probe below the cup. When the probe was positioned at exactly the center of the cup, the results as shown in black were observed. (This scale is also used for representing the temperature versus voltage, graph #4).

Both of these scales become steeper near their upper limits. The probable cause of this is simply electrical resistance. The resistance in the Nichrome wire at these higher temperatures is much greater and therefore of consequence.

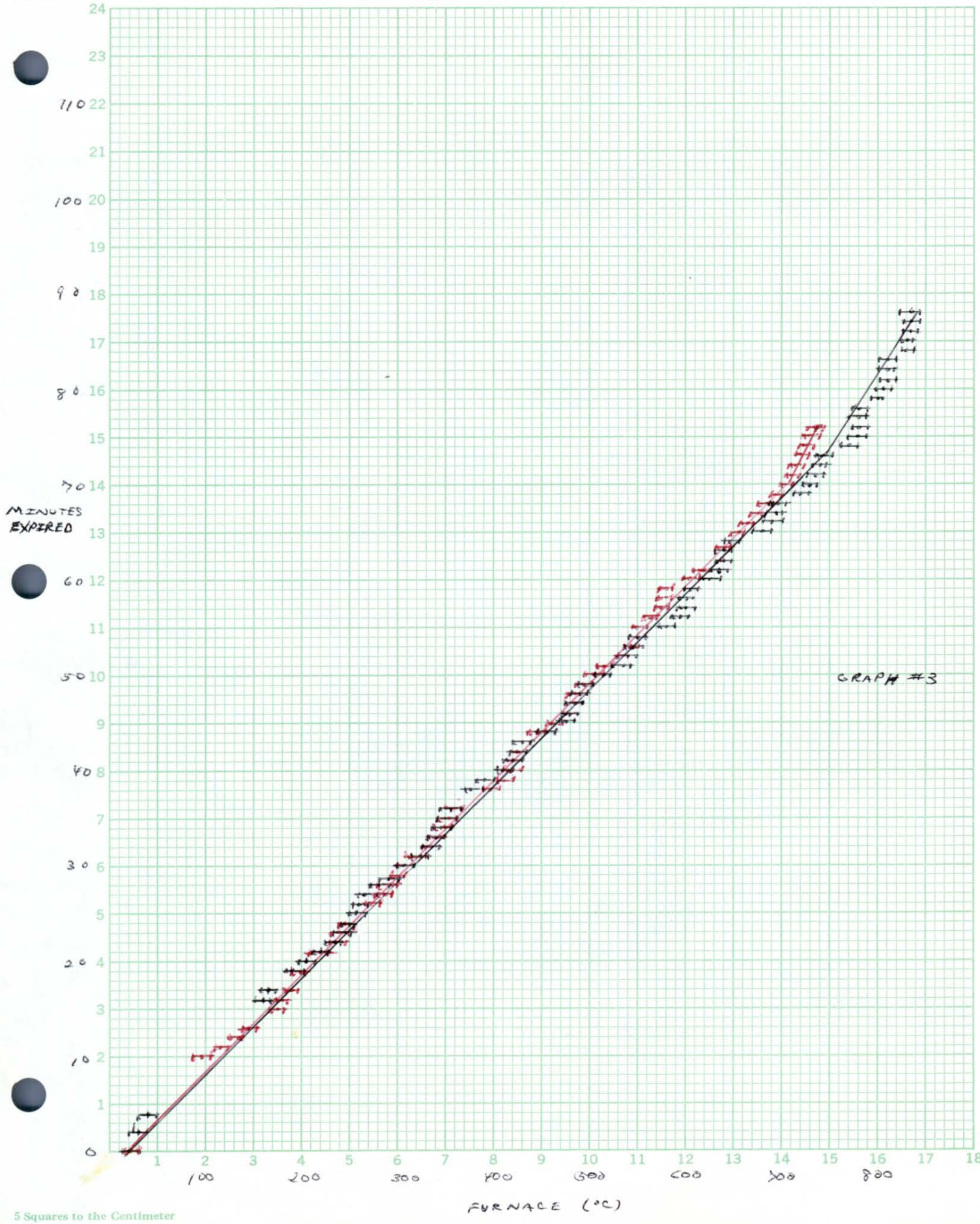
A recurring problem with the furnace that was not remedied until late in the experiment was a short-circuiting of the wiring. Actually, since the heating current is 60 Hz taken from the wall via a variac regulator to the Nichrome wire windings, the increased vibrations with increased A.C. voltage were causing adjacent wires to eventually short. When two tubes of quartz were used to insulate the input-output leads to the top of the coil, the problem was solved.

BALANCE CURRENT
VS.
CONTROL COIL FIELD

5 Squares to the Centimeter

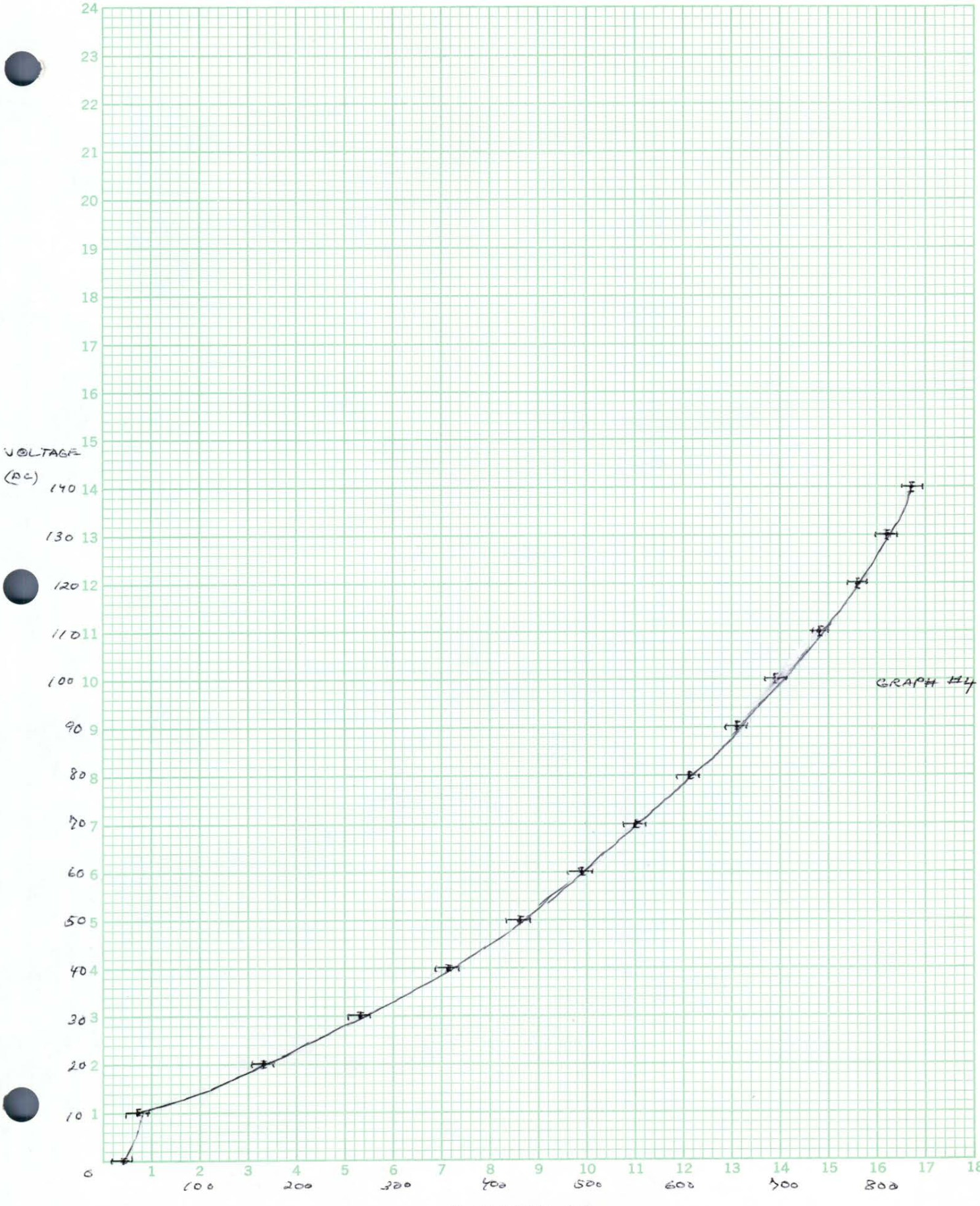

BALANCE
CALIBRATION
(NO SAMPLE IN CUP)
BALANCE CURRENT (i_b) (MA)
POWER SUPPLY (VOLTS)
GRAPH #2
5 Squares to the Centimeter

FURNACE CALIBRATION (TIME VS. TEMPERATURE)



5 Squares to the Centimeter

FURNACE CALIBRATION
(FURNACE VS. VOLTAGE REGULATOR)



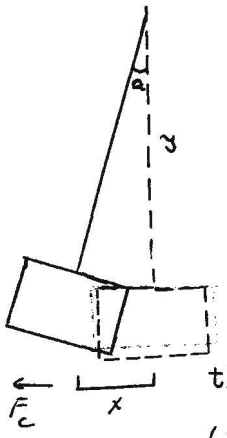
CALCULATION OF THE MOMENT p

and

ANALYSIS OF ERROR

$mga = H_c 2p$ where $m = 24.85 \pm .05$ grams

$g = 980 \frac{cm}{sec}$



$H_c = 1.9 \pm .04$ gauss (250 ma applied through coil; see graph #1)

$a = .125 \pm .107$ rds. ($a = \frac{x}{y}$ where $x = 1.1 \pm .1$ cms.)

$y = 8.7 \pm .1$ cms.)

therefore:

$$\frac{(24.85 \pm .05)(980)(.125 \pm .107)}{2(1.9 \pm .04)} = p$$

and $p = 801.085 \pm 72.53$ emu

The error in all the calculations in this experiment involed the following statistical analysis equation:

$$e_c^2 = \left(\frac{\partial f}{\partial M_1} e_1\right)^2 + \left(\frac{\partial f}{\partial M_2} e_2\right)^2 + \left(\frac{\partial f}{\partial M_3} e_3\right)^2 + \ldots + \left(\frac{\partial f}{\partial M_R} e_R\right)^2$$

In the case of this problem, this becomes

$$e_p^2 = \left(\frac{ga}{2H_c} e_m\right)^2 + \left(\frac{mg}{2H_c} e_a\right)^2 + \left(\frac{mga}{2H_c} e_{H_c}\right)^2$$

The errors in all experimental calculations have been calculated as such. It involves a long and tedious process and need not be shown. However I will list these errors in all results and further calculations.

Equations remastered with the assistance of the Senior Thesis advisor, Hallan C. Noltimier, on 7-10-2014.

SAMPLE CALCULATIONS

The following example was taken from data obtained from the hematite test. (Sample #2)

H_{cr} = .23 ± .04 gauss (field at start; 33 ma applied through coil; see graph #1 and graph for hematite)

H_{cc} = .44 ± .04 gauss (field at curie temperature; 60 ma applied through coil; again, see the appropriate graphs

$F_{cr} = (.23 \pm .04)(2)(801.08 \pm 72.53) = 368.49 \pm 72.25$ dynes

since $F_{cr} = H_{cr}2p$ (force on the coil from the ferrite magnet)

and $F_{cc} = (.44 \pm .04)(2)(801.08 \pm 72.53) = 704.95 \pm 90.45$ dynes

since $F_{cc} = H_{cc}2p = F_o$ at the curie temperature (force on coil at this time)

therefore:

$Wk_t = (.307 \pm .004)\ (336.46 \pm 162.7) = 103.29 \pm 49.96$ dynes

since $Wk_t = \frac{S_2}{S_1}(F_o - F_c)$ where $S_2 = 6.6 \pm .001$ cms

$S_1 = 21.5 \pm .001$ cms

therefore:

since $J(H) = \frac{Wk_t}{v}$ where $v = \frac{1.385 \pm .01 \text{ grams}}{5.26 \text{ theoretical density}} = .263 \pm .002$ cc

and $\frac{H}{y} = 83 \pm 1$ (from graph at rear) gauss/cm

$$J(H) = \frac{(103.29 \pm 49.96)}{(.263 \pm .002)(83 \pm 1)} \text{ gauss/cm} = 4.73 \pm 2.29 \text{ Oersted}$$

and finally

since $k_t = \frac{J(H)}{H}$ where $H = 290 \pm 1$ gauss (from graph at rear)

$$k_t = \frac{(4.73 \pm 2.29)}{(290 \pm 1)} = .016 \pm .007 \text{ (unitless)}$$

The previous set of calculations serves as an example for the rest of the project, since listing every calculation would be a task not worth the space or trouble. In checking any work, one simply can use the previous example as a guide, since it was a consistent technique used throughout the experiment. The results of these calculations are listed on the following pages to simplify their accessibility. The results are graphed with each individual mineral.

CALCULATED VALUES FOR k_t

The following values of k_t are listed with the measured values of H (the electromagnet field) and $\frac{\partial H}{\partial y}$, the field gradient, for convenience. The values for k_t were randomly calculated.

	H gauss (± 1)	$\frac{\partial H}{\partial y}$ $\frac{\text{gauss}}{\text{cm}}$ (± 1)	k_t values 20°C to curie T
HEMATITE			
sample #2:	290	83	.016 ± .007
			.008 ± .007
			.006 ± .007
			.003 ± .007
sample 3:	290	83	.007 ± .009
			.002 ± .009
MAGNETITE			
sample 1	160	32	.832 ± .322
sample 2	160	32	.744 ± .637
MAGHEMITE			
sample 1	290	83	.057 ± .024
			.044 ± .025
			.026 ± .024
sample 2	290	83	.025 ± .020
			.015 ± .020
			.005 ± .020
PYRRHOTITE			
sample 1	200	60	.210 ± .067
			.150 ± .067
			.120 ± .067
			.090 ± .068
			.024 ± .068

	H	$\frac{\partial H}{\partial y}$	k_t values
PYRRHOTITE			
sample 2	200	60	.209 ± .054
			.171 ± .055
			.117 ± .054
			.078 ± .054
GOETHITE			
sample 1	290	83	.020 ± .009
			.014 ± .009
			.008 ± .009
			.005 ± .009
sample 2	215	72	.027 ± .030
			.014 ± .030
			.012 ± .030
			.004 ± .032
LIMONITE			
sample 1	290	83	.018 ± .014
			.012 ± .015
			.010 ± .015
			.006 ± .015
sample 2	290	83	.032 ± .012
			.028 ± .015
			.016 ± .015
			.008 ± .015
PYRITE			
sample 1 1st heating	290	83	.010 ± .040
sample 2 2nd heating	215	72	.098 ± .043

	H	$\frac{\partial H}{\partial y}$	k_t values
PYRITE			
sample 2			
2nd heating (continued)			.057 ± .045
			.030 ± .045
			.013 ± .043
sample 2			
3rd heating	215	72	.253 ± .045
			.189 ± .045
			.013 ± .045
ILMENITE			
sample 1	290	83	.016 ± .016
			.011 ± .016
			.006 ± .016
sample 2	215	72	.009 ± .054
			.007 ± .054

It may be noticed that the values at the curie temperature have been purposely left out. This is simply because these are always measured as zero, and therefore only implied, to prevent redundance. These values have been graphed with the values here, however.

HEMATITE

Fe_2O_3

Hematite is characterized by parasitic ferromagnetism. Yet in order to understand this phenomenon, it is important to briefly describe antiferromagnetism. In ferromagnetic minerals, the magnetization vectors are all parallel and in one direction. In antiferromagnetics, these vectors are antiparallel. This is due to a positive exchange interaction (Nagata, p. 32). Generally, these materials are weakly magnetic. They do take a value of k at a certain temperature that shows abnormally high magnetic properties.

In parasitic ferromagnetism, the spontaneous magnetization disappears at this temperature. This could be because of ferromagnetic impurities or a deviation of the magnetic moment of antiferromagnetic atoms.

Unfortunately, this property cannot be observed completely with the equipment used in this experiment. The particular temperatures at which this can be observed take place at approximately 250°K or -23°C. However, the disappearance of the feeble ferromagnetism which characterizes hematite is visible. In both sample 2 and 3, (1 was incapable of being measured) there is a complete disapperance of magnetic properties between 700°C and 740°C. The actual measured curie temperature is 675°C. The error is easily attributed to (1) the time taken to heat the sample is much greater than the time taken to heat the surrounding furnace and (2) possible impurities in the sample (1) is by far the more dominant. This is true for all the samples tested. Each curie temperature measured is somewhat higher than the theoretical value.

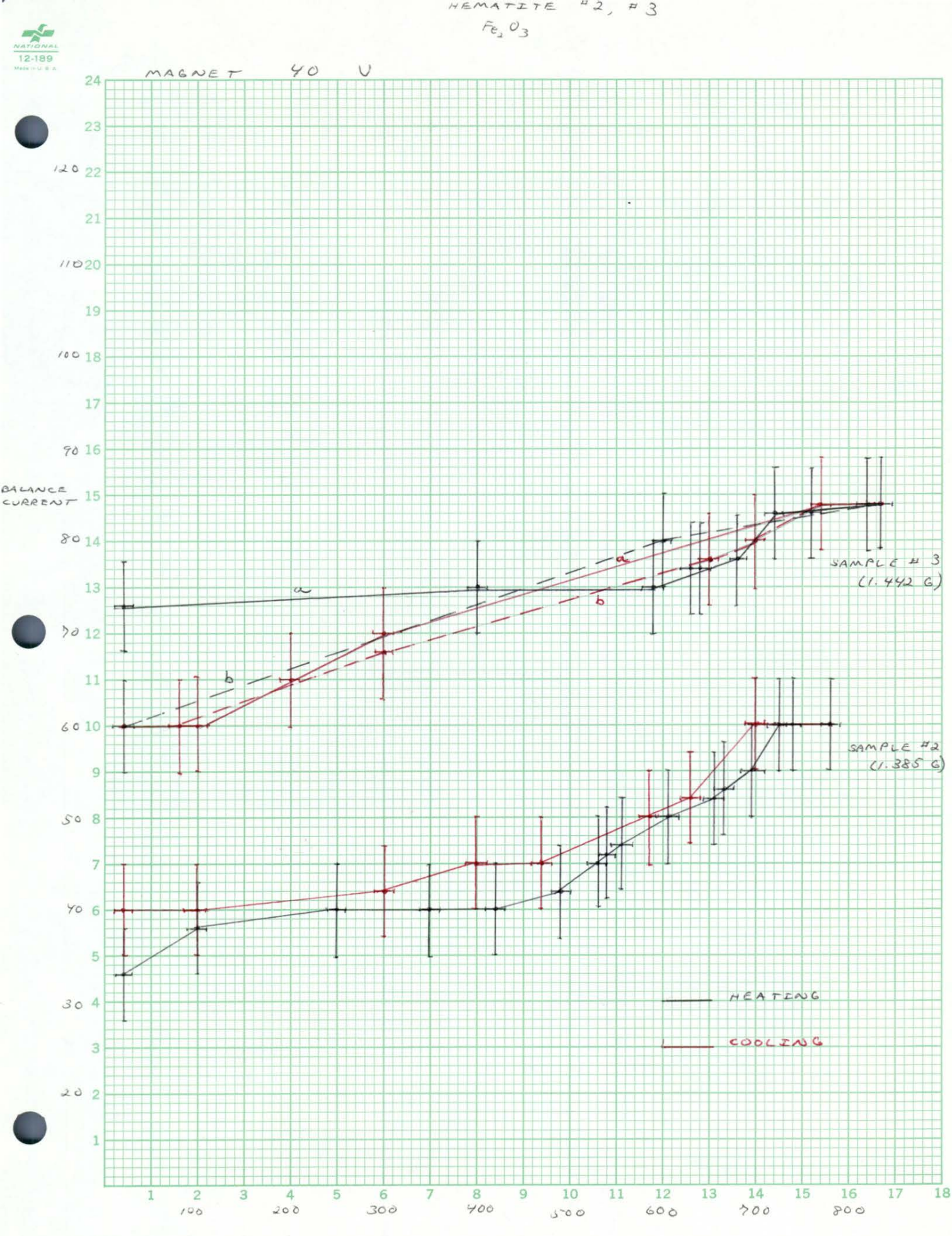
In sample 3 there is a strong deviation following the first heating (a). This can only be attributed to some mechanical failure (such as sticking of the pendulum which did occur at high electromagnet field values). After this (denoted b) the results show an identical scheme as shown with sample 2.

The values of susceptibility calculated are shown in the 2nd following graph. (Sample 3 was calculated from its initial heating a. Its actual value is closer at sample 2). Sample 2 shows a value of (1.6×10^{-2}). Aside from the fact that the error involved seems incredibly large, this is actually a reasonable value, and the error that was calculated was by far larger than needed.

In general, the magnetic nature of hematite is most likely due to the antiferromagnetic causes discussed earlier. In this test, the heating of the mineral either caused magnetic breakdown of the ferromagnetic impurities or the antiferromagnetic spontaneous magnetization disappeared.

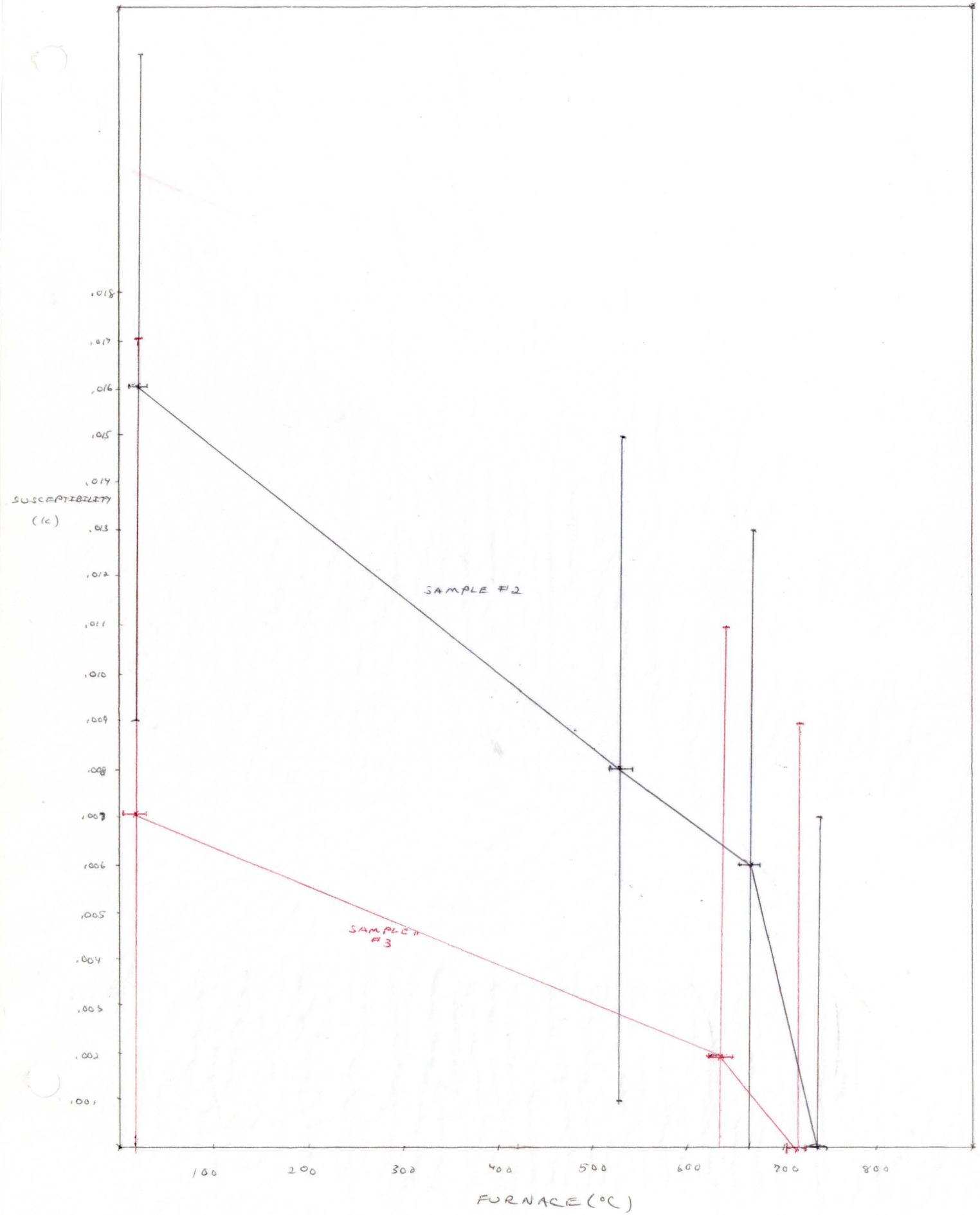
HEMATITE #2, #3

Fe_2O_3



HEMATITE

Fe_2O_3



MAGNETITE

Fe_3O_4

This mineral ushers in a not yet discussed phenonomenon, that of ferrimagnetism. Generally, the ferrites described in chemical composition by MFe_2O_3, where M denotes a valency of 2, show ferromagnetic characteristics. However, upon further analysis, these specimens reveal an antiparallel arrangement so as to appear antiferromagnetic. Yet when the moment of the entire crystallographic structure of the mineral is observed, it appears that the latice has a net moment in one direction, giving magnetic characteristics nearly the same as ferromagnetics. (Nagata, p. 33)

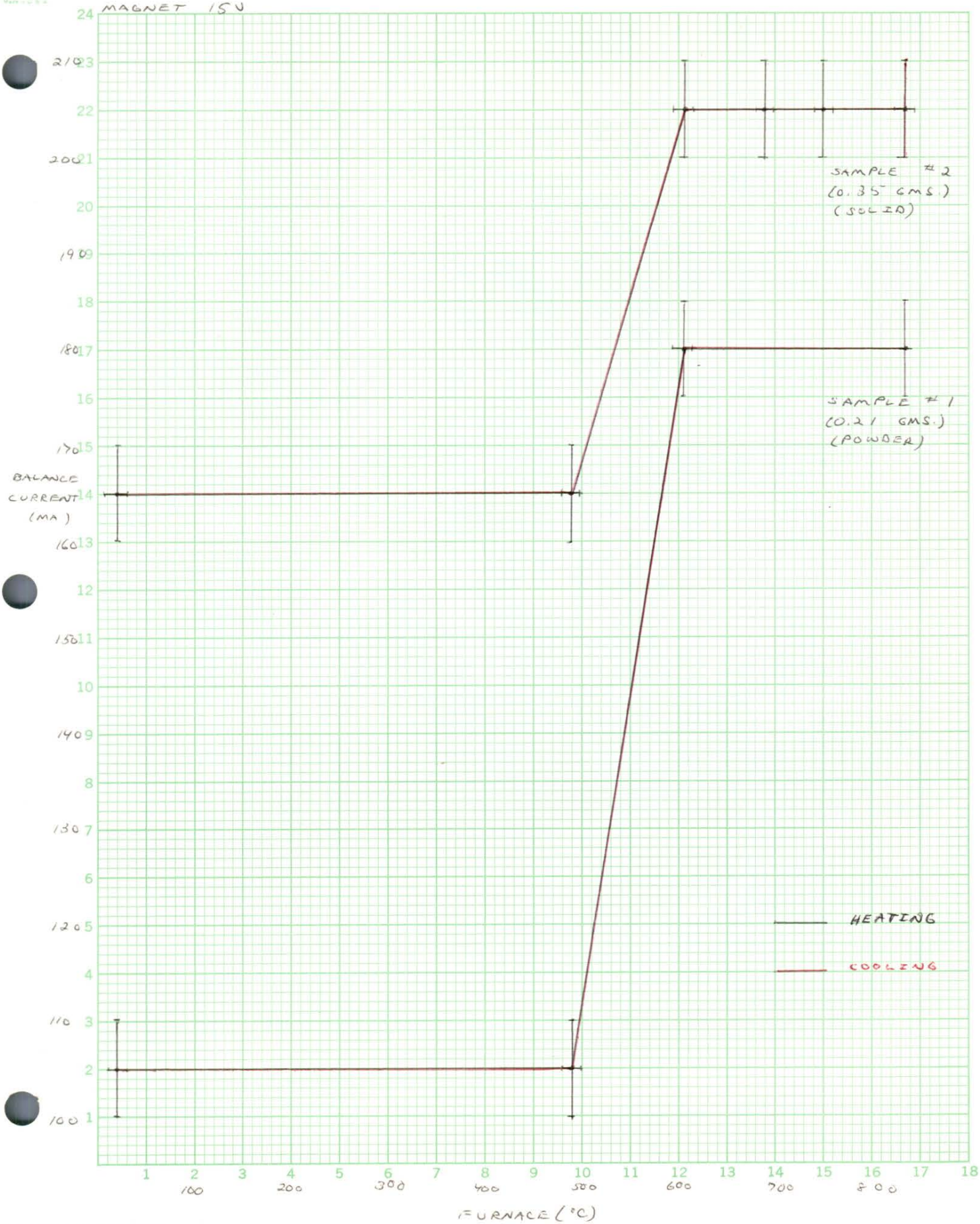
The results shown by magnetite are basically predictable. A rather large value for susceptibility at room temperature will follow a uniform decline in value with a rising temperature until it is nothing at 575° C. (The value obtained in these results was 605°C, attributable to the time lag in heating the sample as compared to the furnace.) One interesting characteristic of the two samples was the larger displacement exhibited by sample 1. This specimen was a pulverized mass from a different source rock than 2. Again this was probably due to impurities and (or) a slightly differing chemical composition. Although the powder may have been more thoroughly heated than the solid in the time allotted since this time was equal.

This mineral was run before the attachment of the light source, largely because its magnetization changes are easily visible. The result is that small deflections encountered in heating prior to tne large deviation at the Curie point could not

be seen. To that may be added the fact that above the curie temperature ferrimagnetics do not behave as normal ferromagnetics, however this change is dependent on chemical composition, and is very minute as a result. Hence this characteristic was not detected in any ferrimagnetic specimen tested.

MAGNETITE
Fe_3O_4

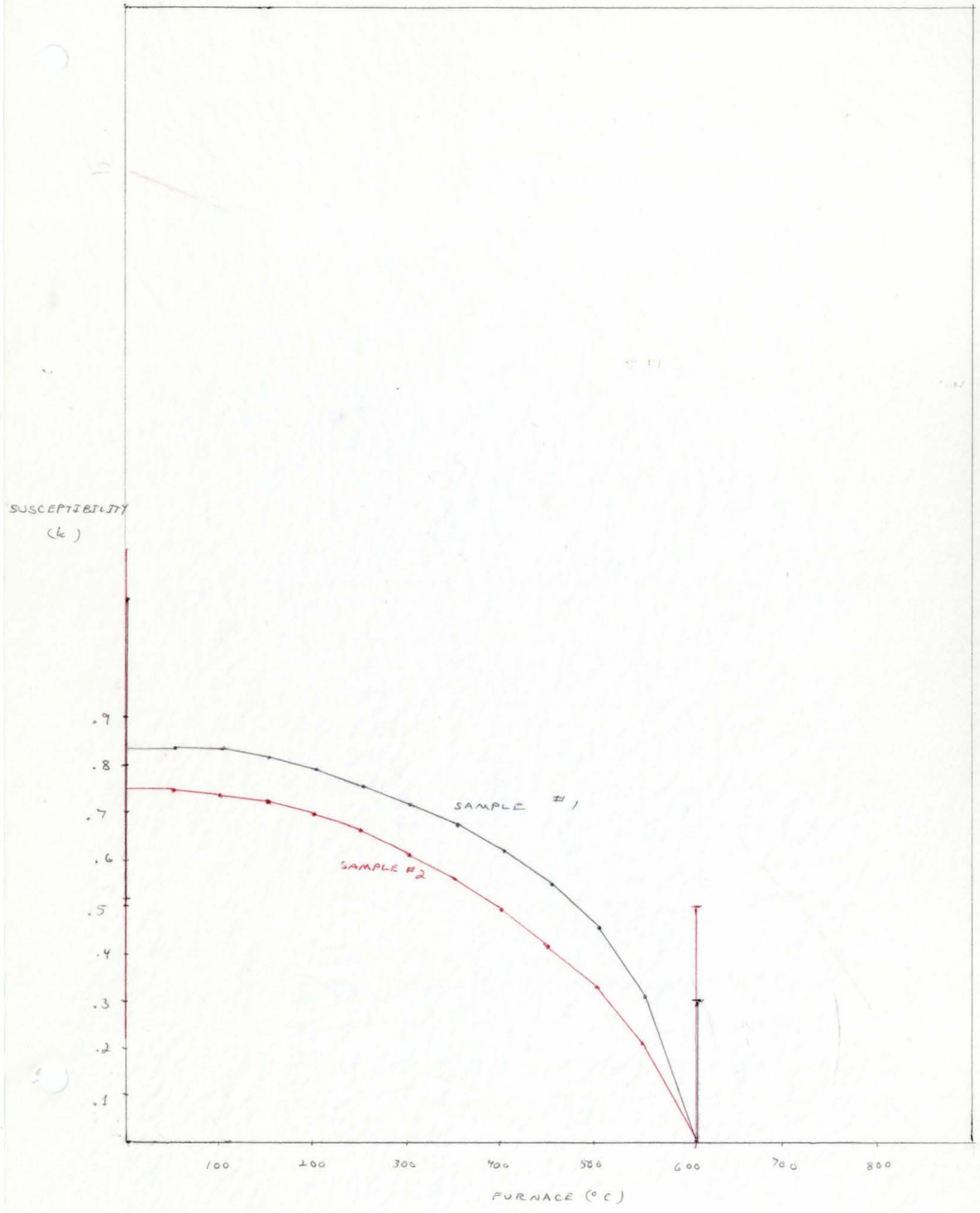
MAGNET 15V



5 Squares to the Centimeter

MAGNETITE

Fe_3O_4



MAGHEMITE

γFe_2O_3

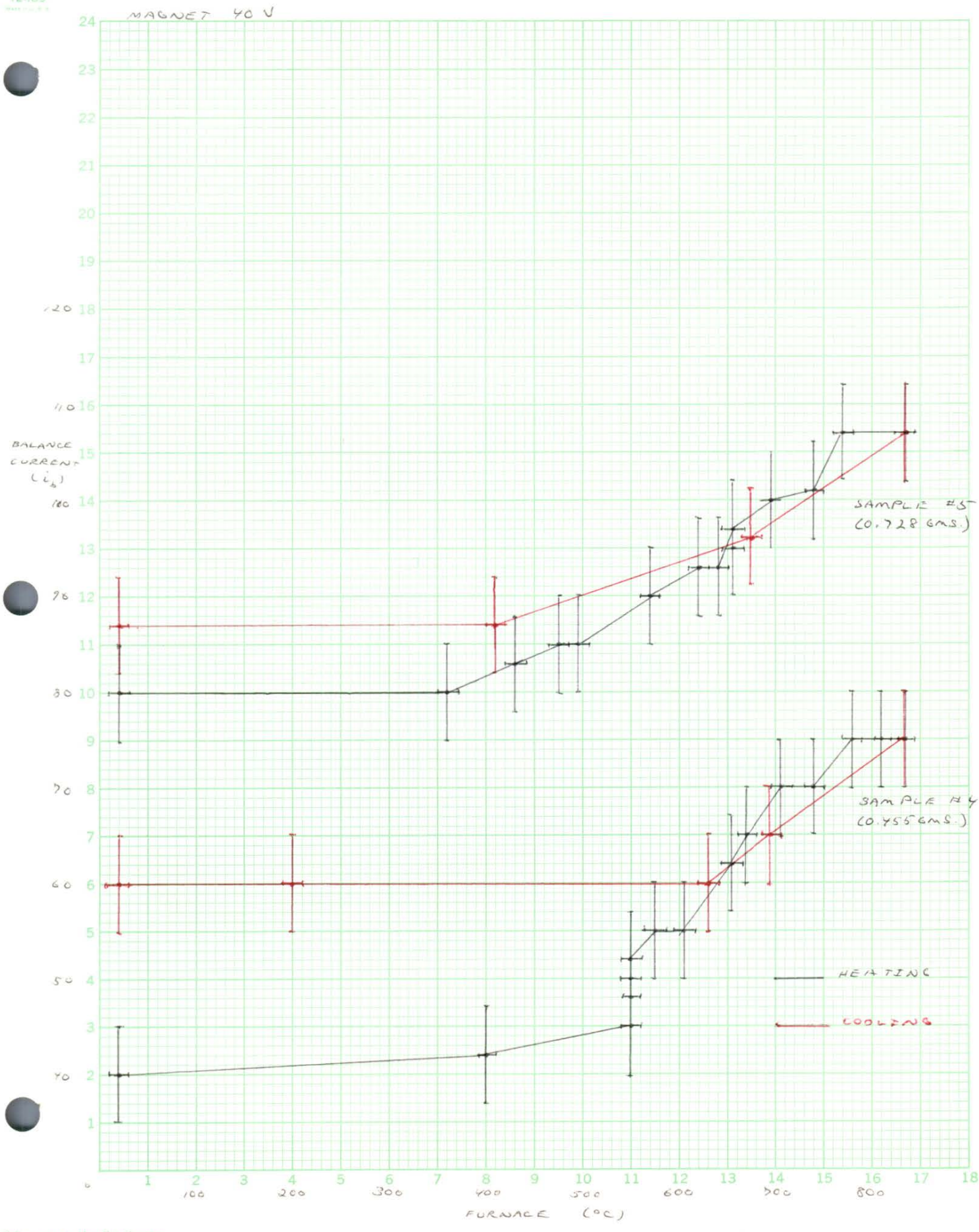
Maghemite is similar to magnetite in crystallographic structure, yet is characterized by certain cation vacancies. It is very unstable on heating and readily reverts to hematite. This temperature has been shown to be near 275°C, and as a result the curie temperature has never been accurately described. (Strangway, p. 469) It has been extrapolated from solid-solution parameters, however, between maghemite and magnetite. The value given for this temperature is 675°C. (Nagata, p. 85.)

In the data from this experiment a conversion point is demonstrated with both samples. However the values of the susceptibility that were calculated were much too low than theoretical values permit. This can be proven by simply observing the pyrite sample tested, which itself converts to maghemite upon heating. Maghemite generated in that test showed a susceptibility nearly a power of ten larger than that calculated with the purported maghemite examined here (0.26 compared to .03 and .06).

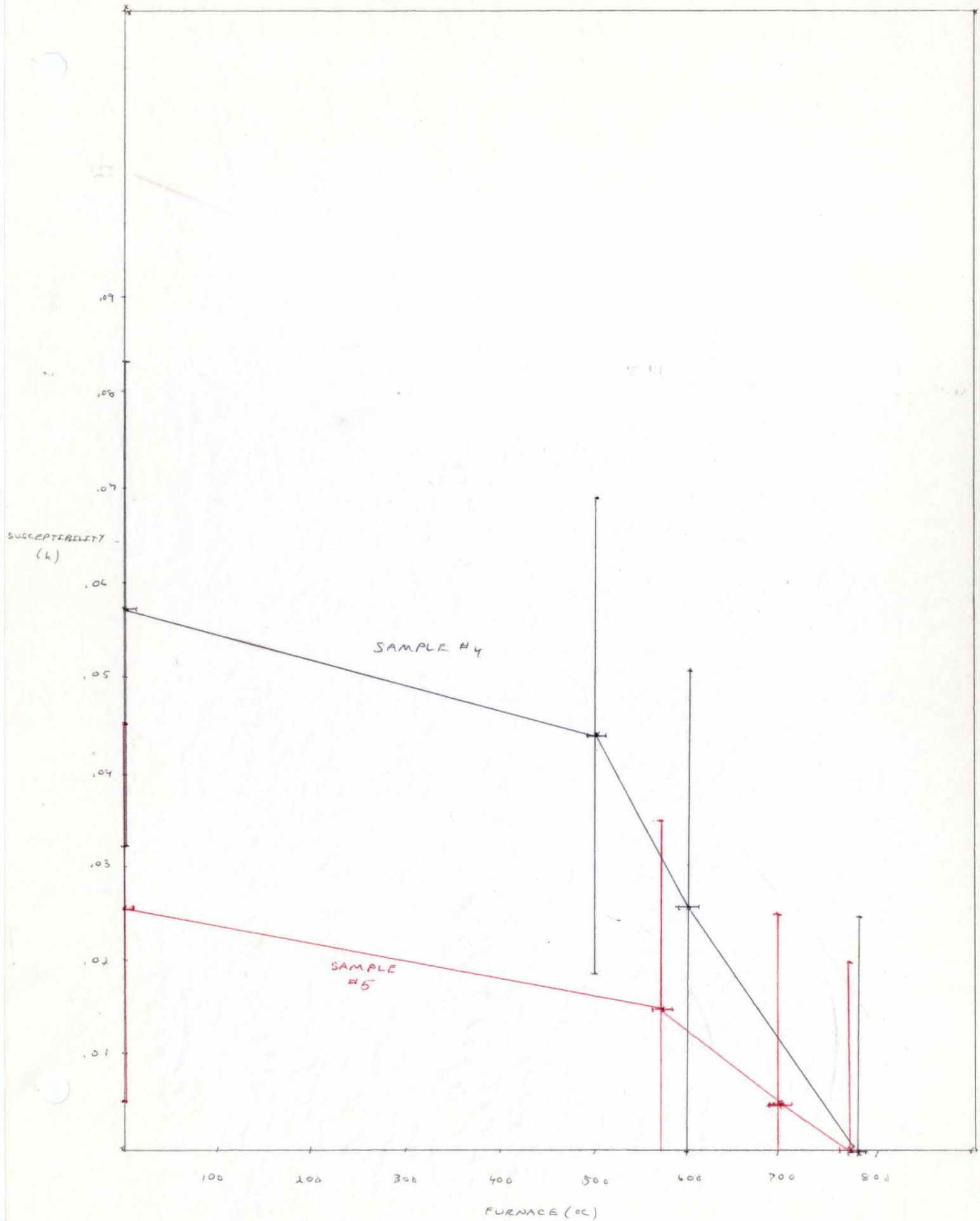
No doubt some conversion is taking place, but either that conversion is incomplete or the sample is extremely impure and not true maghemite. The later seems more formidable, for the sample was heated for over two hours. The susceptibility curve is generally smooth, although it would be more accurate to show a double lobed curve as is demonstrated with limonite and goethite. The reason this was not done was simply because an accurate conversion temperature could not be verified simply by the data given on the heating-cooling graphs. It can be noted that the supposed

conversion temperatures exhibited by both samples differs by nearly 200°C. Again, most probably impurities in the latice of the source rock is the probable cause.

MAGHEMITE
γFe_2O_3



MAGHEMITE
γ Fe_2O_3



PYRRHOTITE

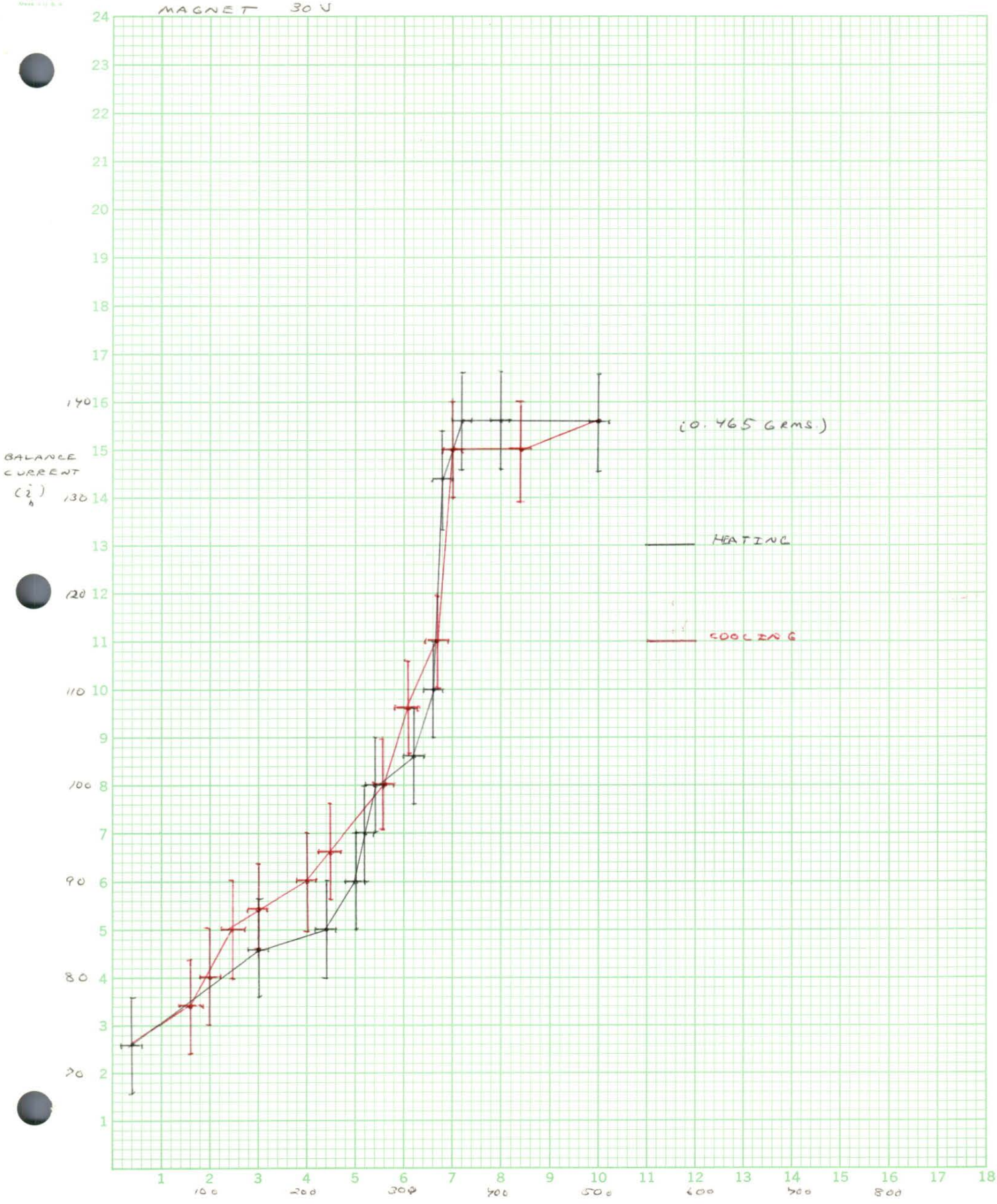
$Fe_{1-x}S$

The obviously magnetic pyrrhotite is a ferrimagnetic mineral. Although both antiferromagnetic specimens have been studied according to the sulphur content (that is to say that a more accurate chemical formula would be FeS_{1+x} (Nagata, p. 119)), we will suffice it to say that this specimen does obey typical ferrimagnetic laws observable by the balance. One can easily check the curves to verify this. As is the case with maghemite, it is a consequence of the iron vacancies filled in by the anions (in this case, the sulphur) that explains its ferrimagnetism and its strongly magnetic characterics.

With both samples examined the data is true to theory. A fairly strong susceptibility(0.20) at room temperature shows complete degeneration at a curie temperature between 310°C and 360°c (sample 1&2). What is remarkable about this specimen was that the time involved in heating was very rapid. The curie point is practically true to theory (340°C) and not high by over 50°C as is the case with most of the other samples. This could be because the sulphur-iron composition is not very heat resistant. As heating increases, sulphur is quickly boiled off as SO_2. (This same occurence, as it will be later shown, is found with pyrite.) The total time elapsed of general heating and cooling was 75 minutes. Nonetheless, it is a mineral simple to analyze within the capabilities of the balance.

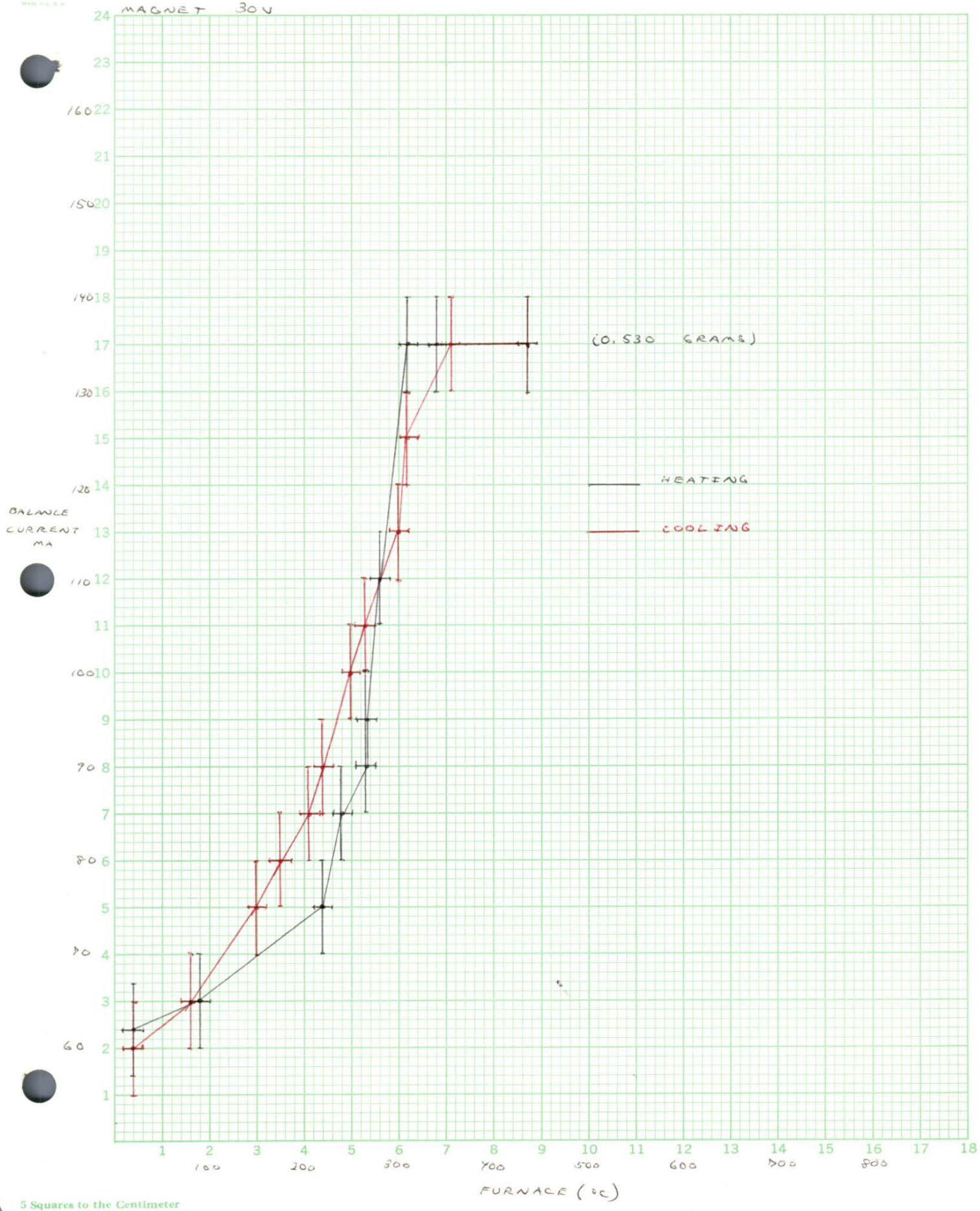
PYRRHOTITE #1
$Fe_{1-x}S$

MAGNET 30 V



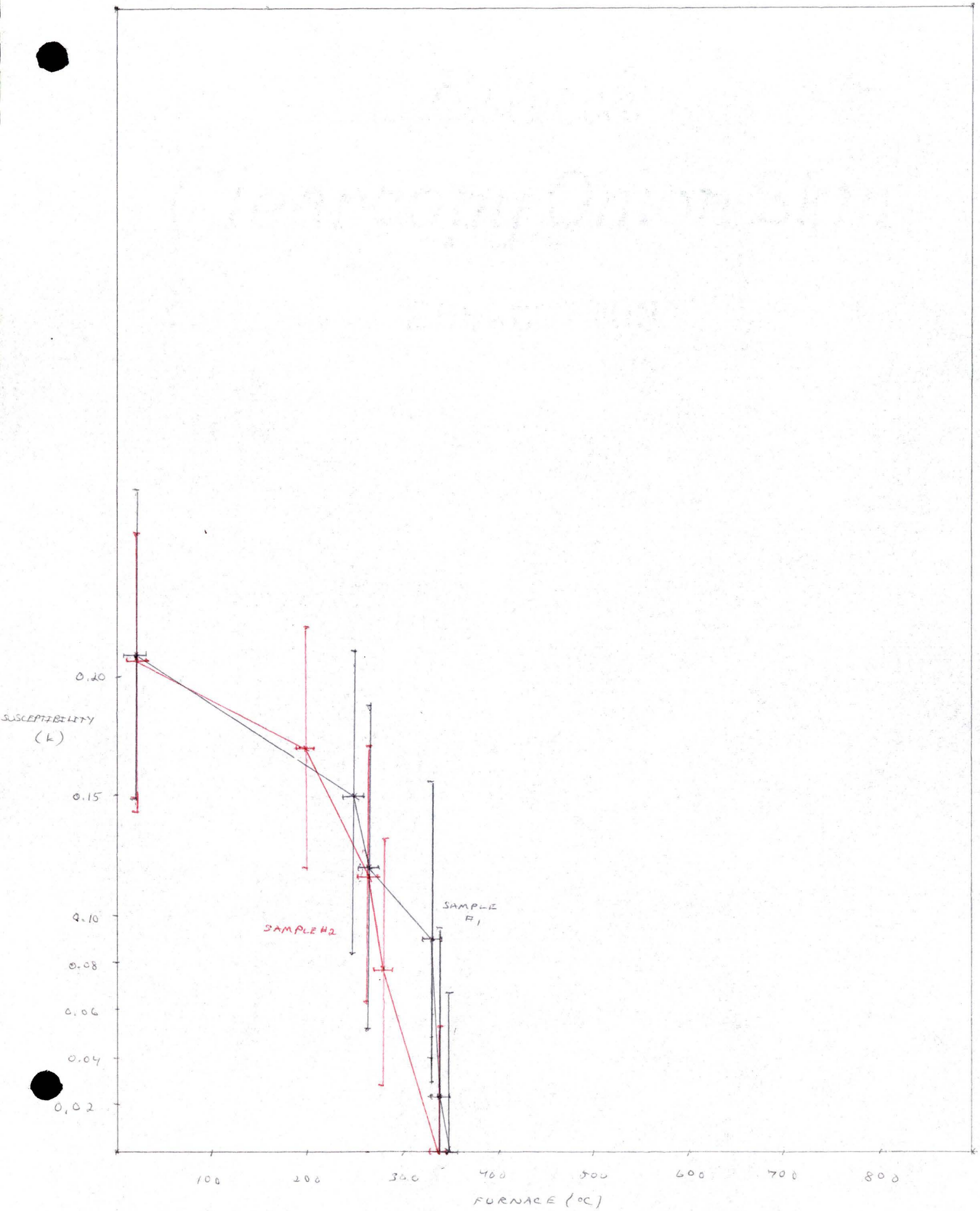
5 Squares to the Centimeter

PYRRHOTITE #2
$Fe_{1-x}S$



PYRRHOTITE

$Fe_{1-x}S$



GOETHITE and LIMONITE

$HFeO_2$ $FeO(OH)\cdot nH_2O$

These two are grouped together for the obvious reasons that they are chemically and crystallographically similar and show magnetic similarities as well. The most striking phenomenon is none but the double-lobe nature of the susceptibility curves for both minerals. Although little information could be found as far as paleomagnetic performance is concerned for these specimens, it can be generally understood that both are degenerating to hematite upon heating. This conversion is marked at the joints of these lobes. (Both lobes are discontinuous beyond this point since it is only an extrapolated value.)

Explaining this conversion can be simply stated as a process of dehydration. With goethite, a hydrate, the conversion occurs rather quickly. Sample 2 follows this pattern, with a marked change near 200°C, (theoretical value: 120° C) and continuing upward as hematite until its curie temperature. With sample 1 the change is also evident, visible at a considerably higher temperature of 350°C. This sample, it can be seen, fell below the cooling curve at 550°C. This is probably a result of the sticking phenomenon that will be explained at the end of the paper. Nonetheless, the value more reasonable is given by sample 2, despite its large error.(This error was only calculated because the magnet voltage and hence the intensity of H was reduced, lowering the efficency of the system. It still seemed efficient enough.)

Limonite has a considerably higher conversion temperature.

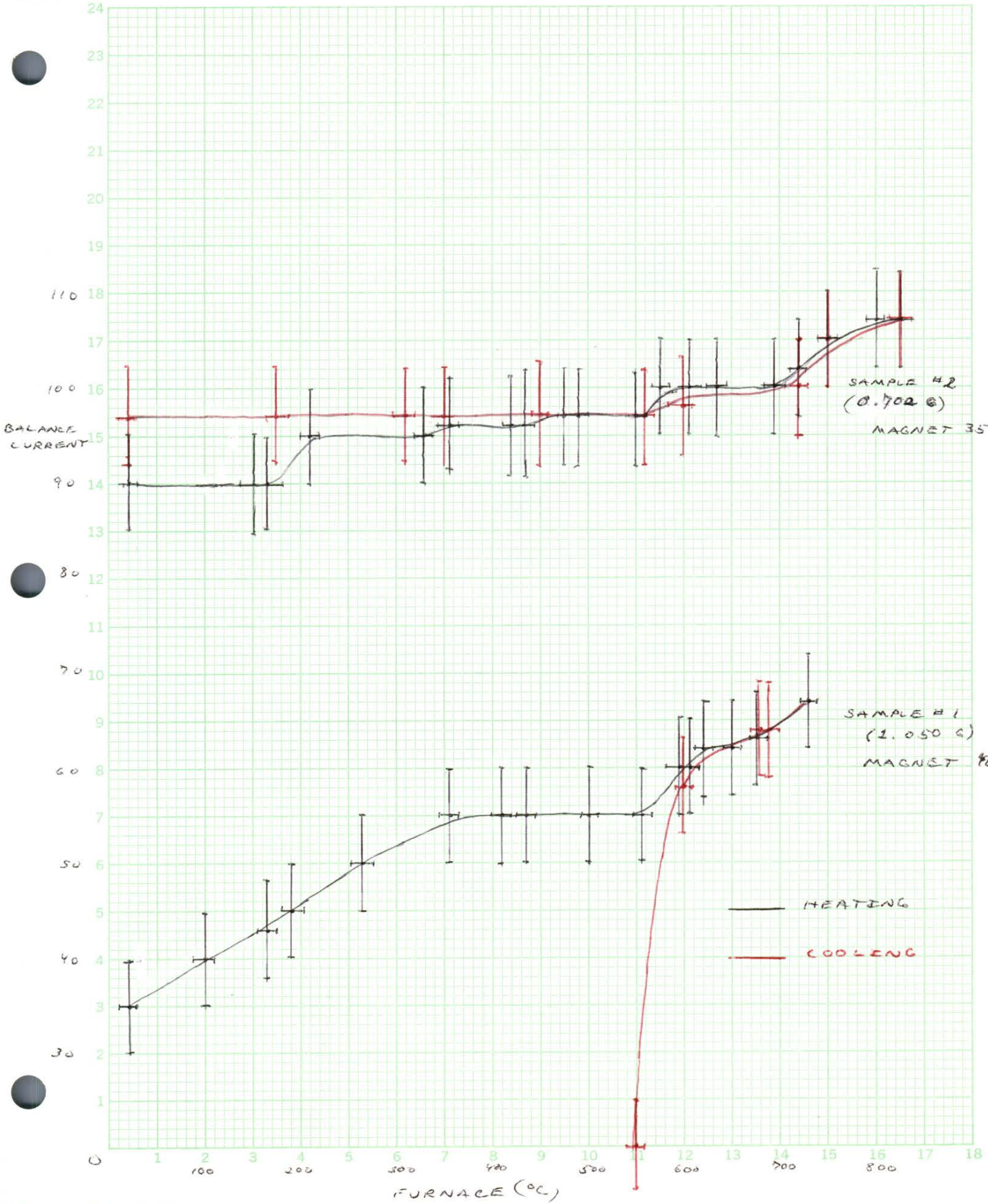
It is not a hydrate but an hydroxide, and takes a greater degree of heating to completely dehydrate and convert to hematite. It is a much more weathered rock than goethite, representing actually a later stage of weathering for goethite. Because of this, the conversion does occur at a higher termperature value. The curie temperatures shown are the measured values for hematite, sample 2 slightly higher than sample 1, but within a normal range possible for this apparatus. The value extrapolated for the conversion of 2 is probably too high, but is nonetheless fitting the geometry of the pattern.

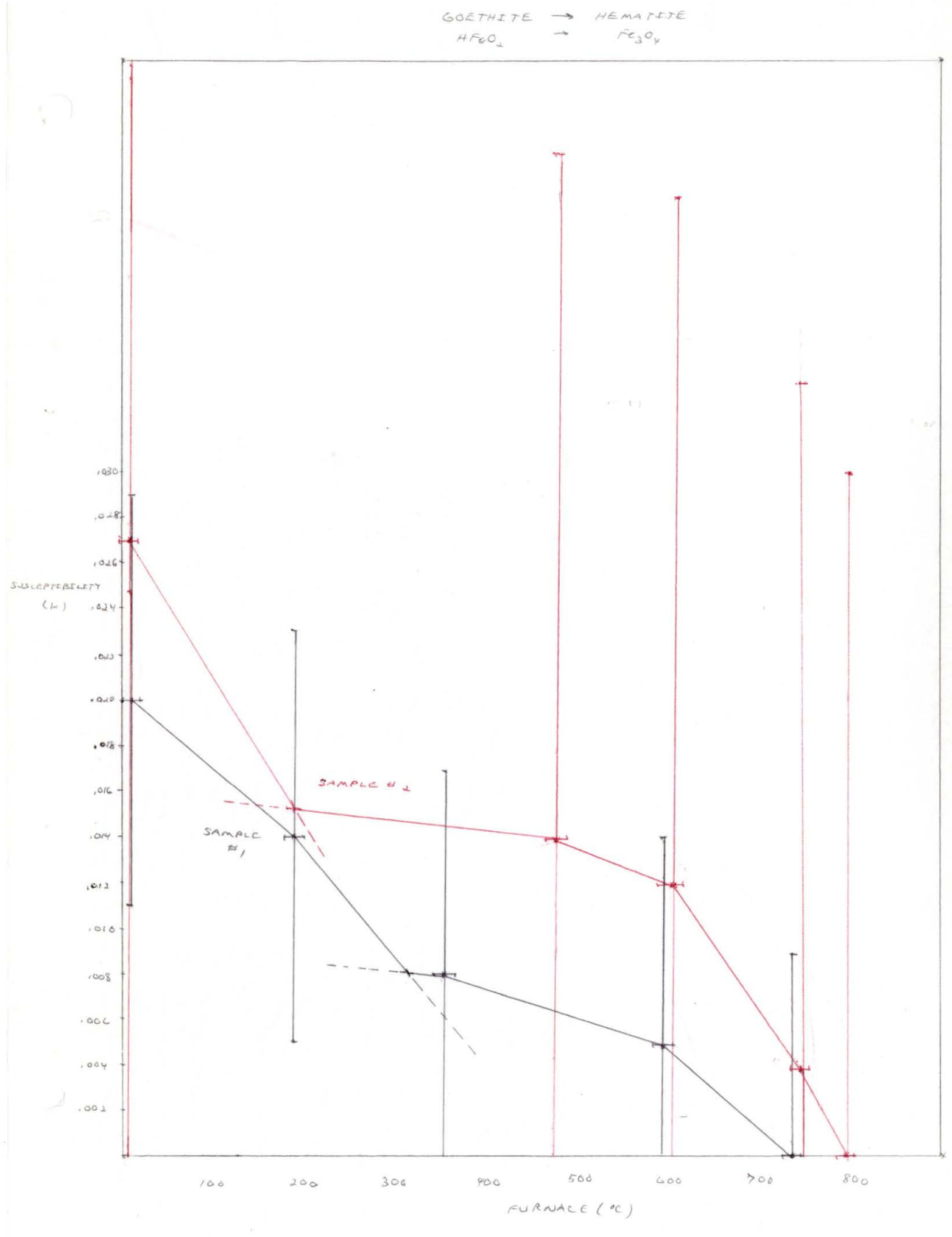
Generally, the limonite susceptibility range is not as definitive as that of goethite. It can be surmised that it has much to do with the hematite generated. Goethite showed a greater degree of weight loss (approximately 8-10%) after heating, as compared to limonite (2-4%). Likewise the conversion was not as rapid for limonite as it was for goethite and therefore the susceptibility fluxuations could not have been easily detected.

It is possible that heating and than cooling below the conversion temperature could show a weak magnetization and therefore a greater susceptibility (Strangway, P.462). Although it was not attempted here for there was tenuous control of heating at such low values (the error becomes too large), the nature of this phenomenon can be explained by ferrogmagnetism. Likewise, it is possible that limonite could do the same.

GOETHITE #1, #2

$HFeO_2$

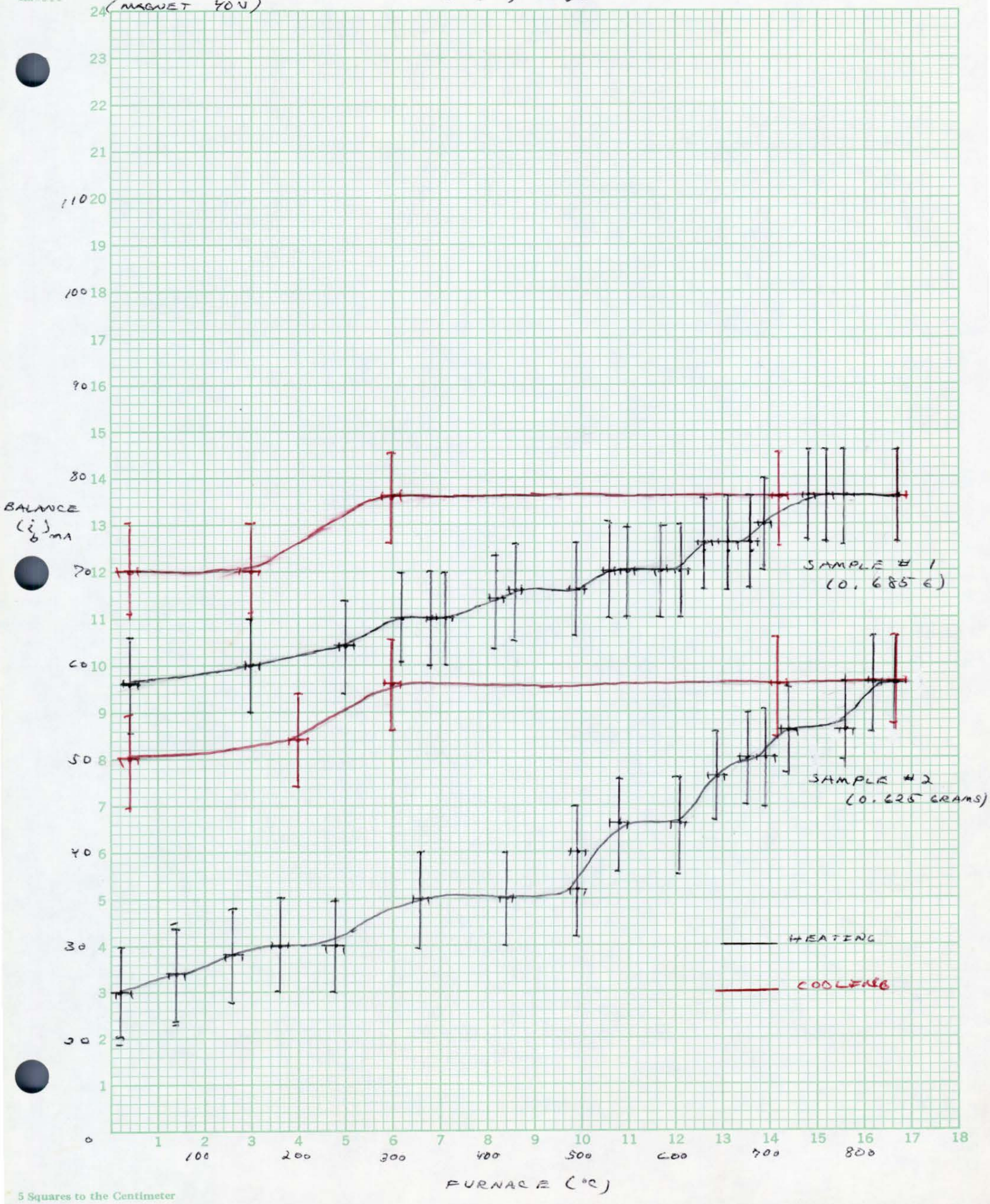



GOETHITE → HEMATITE
$HFeO_2$ → Fe_3O_4
.030
.028
.026
SUSCEPTIBILITY (K)
.024
.022
.020
.018
.016
.014
.012
.010
.008
.006
.004
.002
SAMPLE # 2
SAMPLE #1
100
200
300
400
500
600
700
800
FURNACE (°C)

LIMONITE #1, #2

$FeO(OH) \cdot xH_2O$

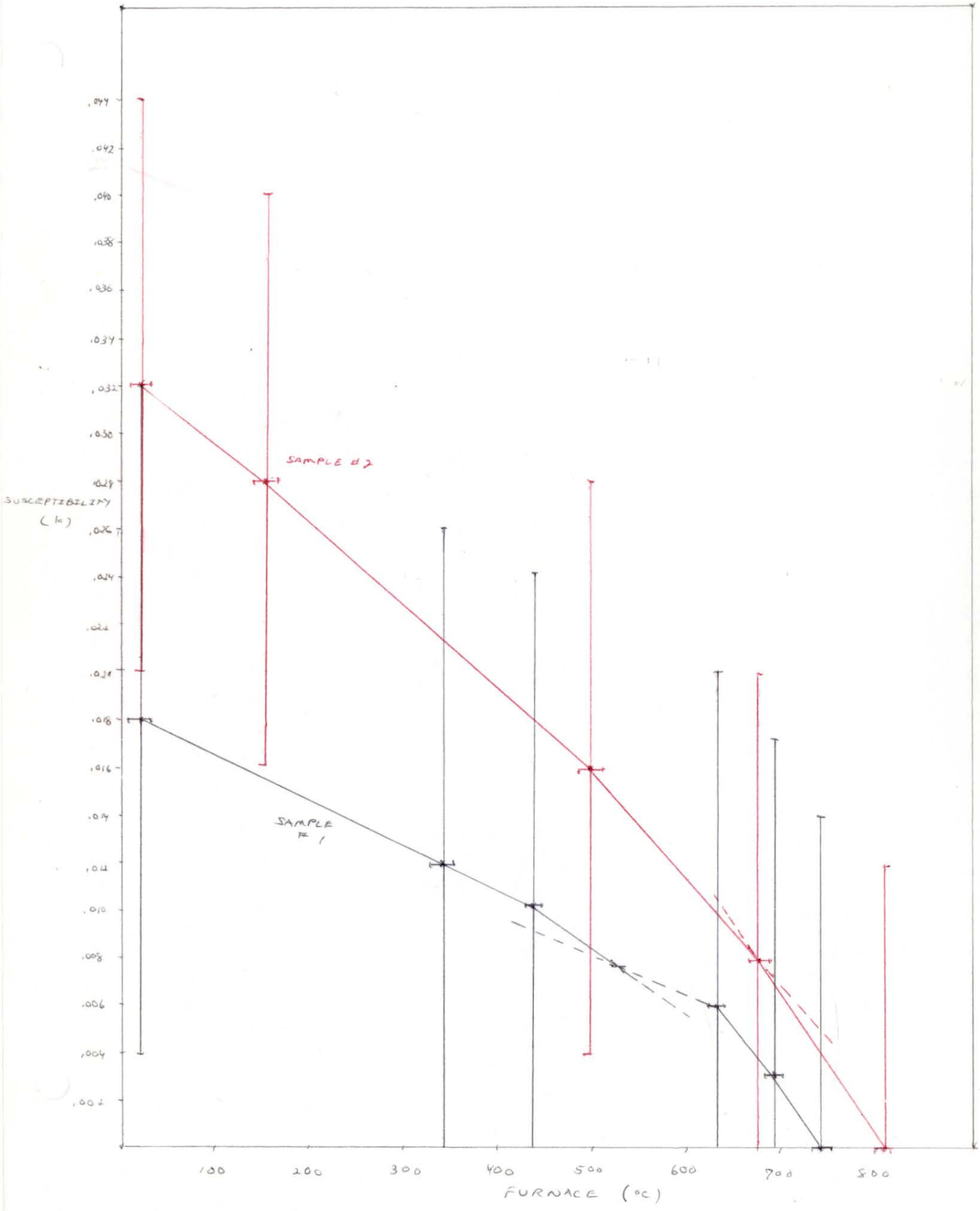
(MAGNET 40 V)



5 Squares to the Centimeter

LIMONITE

$FeO(OH) \cdot nH_2O$



PYRITE

FeS_2

This specimen proved to be perhaps the most interesting of all minerals tested during the entire experiment. Basically, pyrite is nonmagnetic, but upon heating, the sulphur is boiled off in the form of SO_2, leaving a cubic Fe_2O_3, or γFe_2O_3, maghemite. The reason that maghemite is formed and not hematite is that the cubic nature of pyrite is retained longer than the oxidation of the sulphur. Hence maghemite is formed. Naturally, applying continued heating, one will get hematite, as is the case with this sample. (No. 2)

The increased magnetic intensity is seen upon the evolution of maghemite with both samples. In sample 1, the cooling curve dropped below the x-axis, because the scale of magnetization had not been comprehended. (A value for susceptibility had been calculated for sample 1, and this it can be seen is approximatetly 0.1; however, the error is stretching completely off the page. This value is probably much closer to zero.) The theoretical value for the pyrite to maghemite breakdown is 480°C. By the cooling-heating curves, this can be verified. On both samples, the cooling curve departs from the heating curve at 500°C. for both. Again, as is the case with pyrrhotite, the value is not more than 50°C larger simply because SO_2 is boiled off very quickly, and there is little temperature lag between the furnace temperature and that of the sample.

As is indicated by the graphs, the extrapolated point of susceptibility breakdown is much to low. This is simply because

the sample was erroneously assumed to be completely converted to hematite, for as it is seen, the final cooling of sample 2 does not return to its original position it assumed as pyrite, as it should. This however does not affect the values calculated for maghemite.

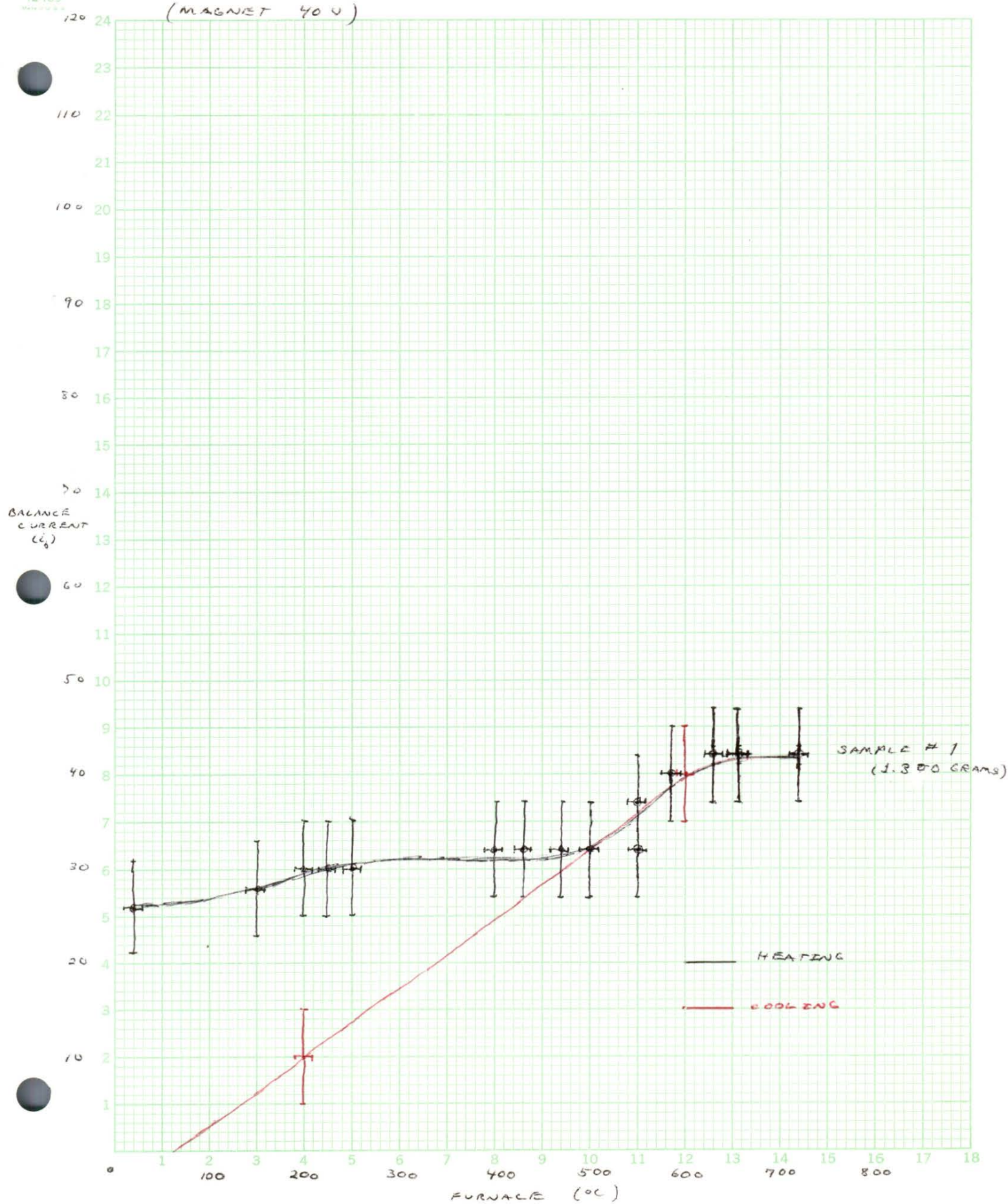
The quantity of susceptibility calculated for the third heating of sample 2, is a much more realistic value than that calculated in the original maghemite run. This sample was heated for a lengthy amount of time (total:3.3 hours), hence it may seem that the original maghemite was not as impure as was thought. Yet the values of susceptibility here are calculated from the beginning of each reading, and are not dependent on the entire duration of heating, but only the duration up to that point. The time duration calculated from the beginning of the test till the end of the 2nd heating (giving the reference for the largest value of susceptibility), was 2.2 hours, almost identical for the heating duration for the original maghemite.

Pyrite must obviously be baked for a lengthy time,much longer than the time allowed here, in order for a complete maghemite-hematite conversion to take place. Just what this time duration must be, to the author,remains a mystery.

NOTE: To prevent confusion, a short explanation of the interpretation of the following curves is offered. The first heating-cooling curve is sample #1. The following three are successive heatings of sample 2, and can be followed in sequence. Likewise, the susceptibility curve for sample 2 is represented by the red, for the 2nd succesive heating of this sample, and the black, for the third heating.(The susceptibility for #1 is on the preceeding page.)

PYRITE FeS_2

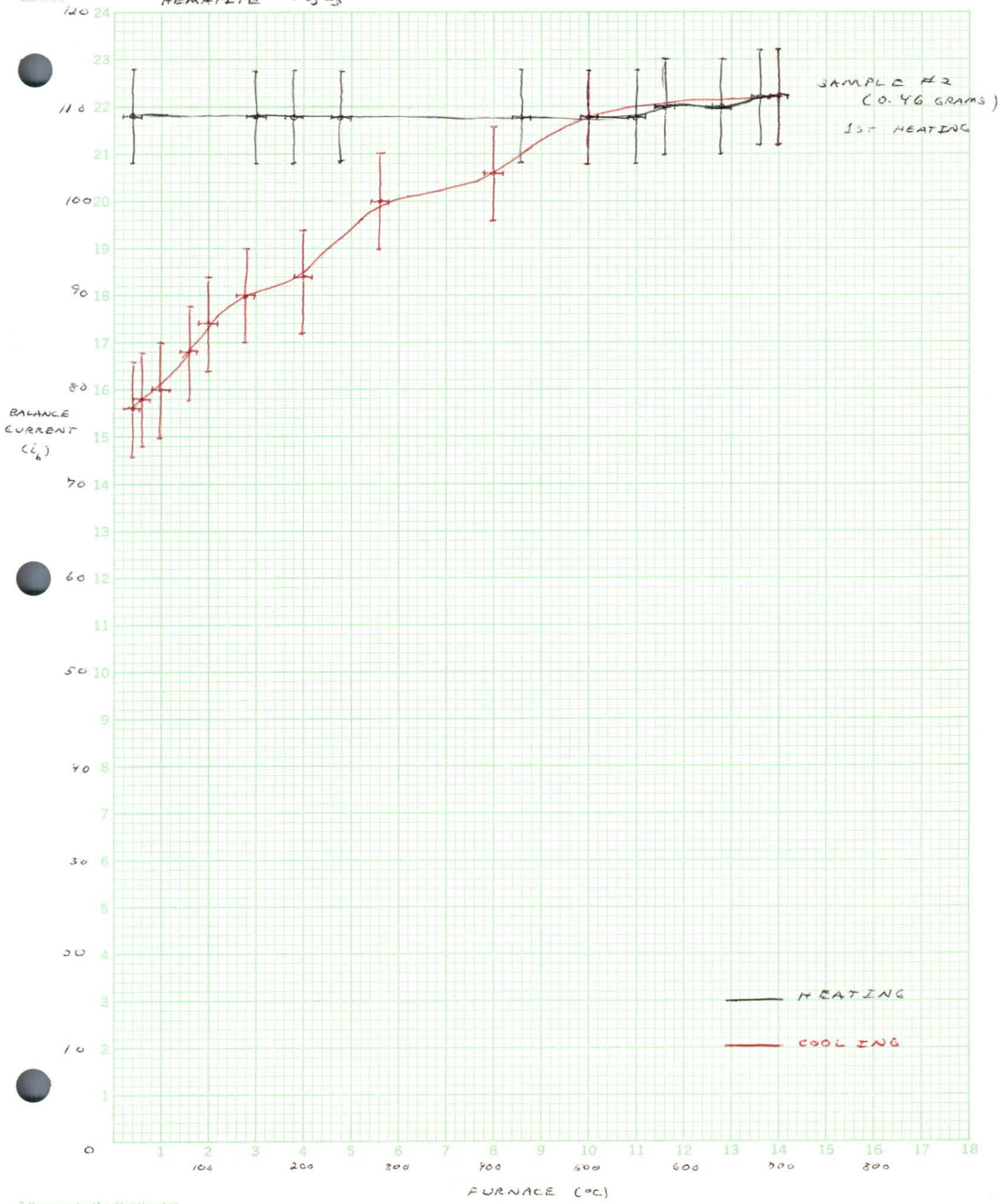
(MAGNET 40 V)



PYRITE #2 FeS_2 MAGNET 35° (≈ 1 AMP)

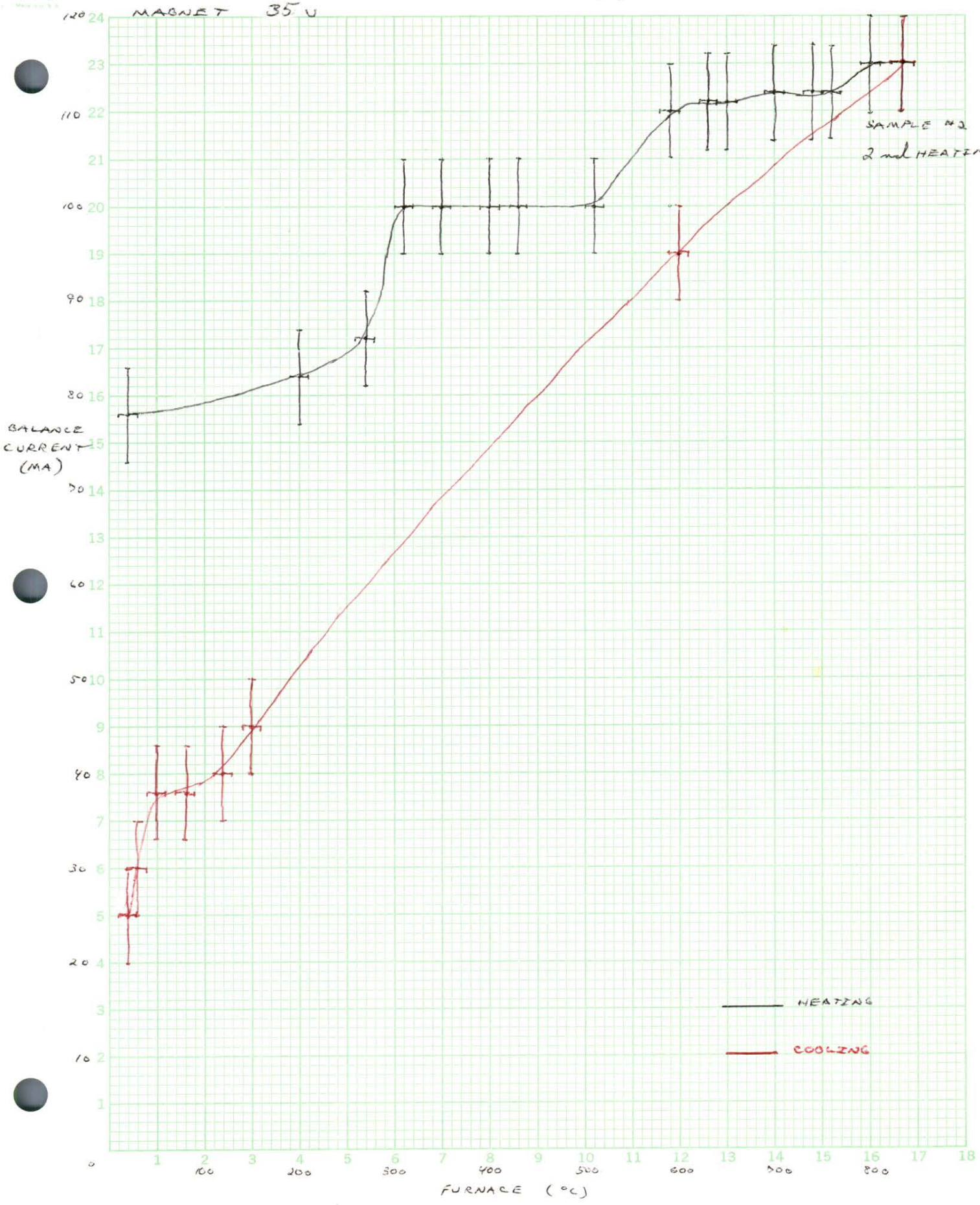
↓ MAGHEMITE γFe_2O_3

↓ HEMATITE Fe_2O_3



PYRITE → MAGHEMITE → HEMATITE

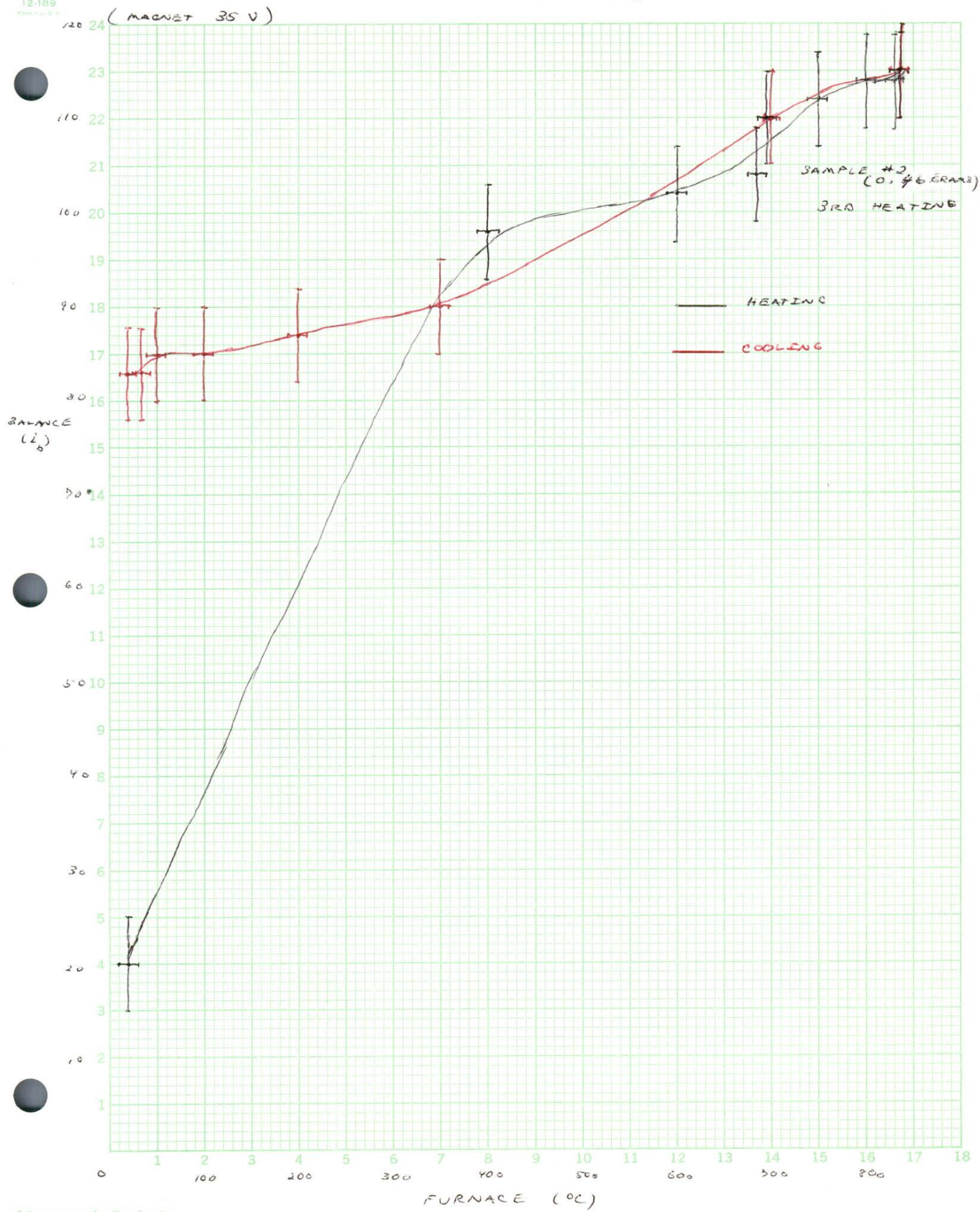
$FeS_2 \rightarrow \gamma Fe_2O_3 \rightarrow Fe_2O_3$



5 Squares to the Centimeter

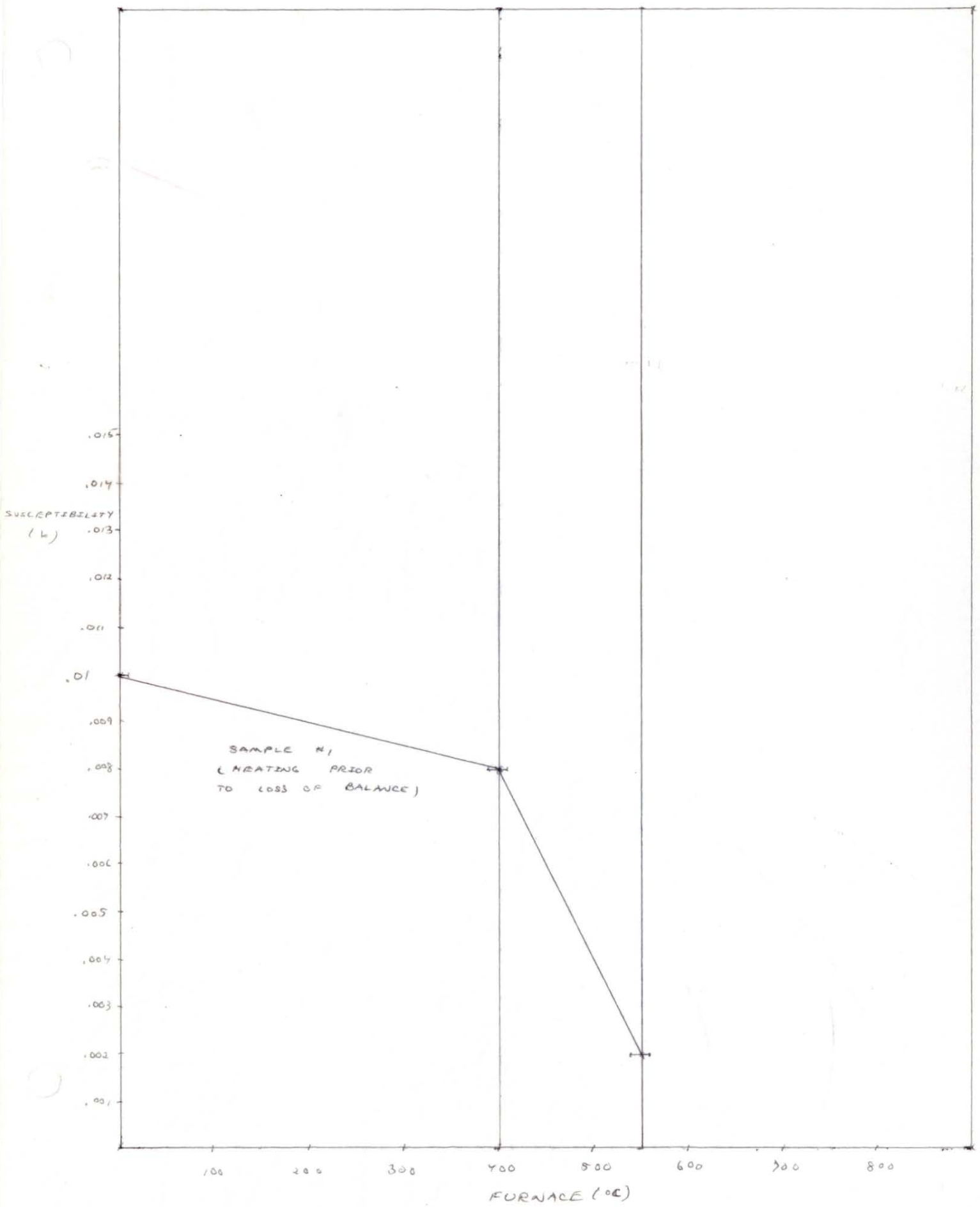
PYRITE - MAGHEMITE

$FeS_2 \rightarrow \gamma Fe_2O_3$



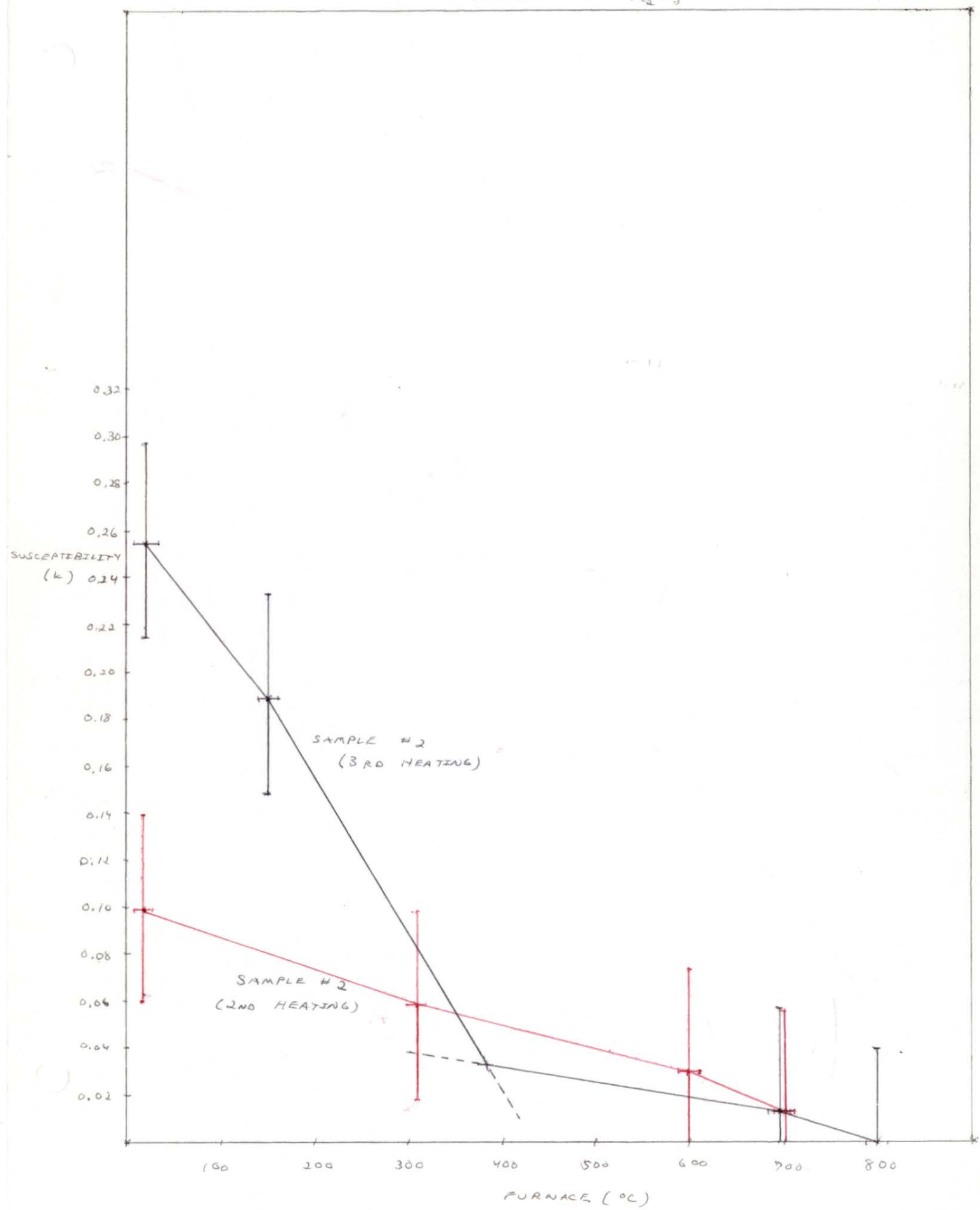
5 Squares to the Centimeter

PYRITE
FeS_2


.015
.014
SUSCEPTIBILITY
(k)
.013
.012
.011
.01
.009
.008
.007
.006
.005
.004
.003
.002
.001
SAMPLE #1
(HEATING PRIOR
TO LOSS OF BALANCE)
100
200
300
400
500
600
700
800
FURNACE (°C)

PYRITE → MAGHEMITE → HEMATITE

FeS → Fe_2O_3 → Fe_2O_3



ILMENITE

$FeTiO_3$

This is perhaps an anticlimactic ending to an otherwise glorious experiment. it is a good example of an antiferromagnetic mineral, but its antiferromagnetism can only be measured at temperatures similar to those approaching liquid air. Otherwise it is only slightly magnetic above these temperatures. (At 68°K, the susceptibility scale will increase over ten times its room temperature value.(Nagata, p. 30))

In procurring a sample of this, the best of two was used from the extensive collection found in Orton Hall. The results show most likely hematite, and not ilmenite. Natural ilmenite has a curie temperature near 350°C, but this sample does not breakdown until 650°C to 700°C (samples 2 and 1 respectively). The susceptibility values as well are nearly identical to those calculated for natural hematite. (.01 and .02 for samples 2 and 1, respectively.)

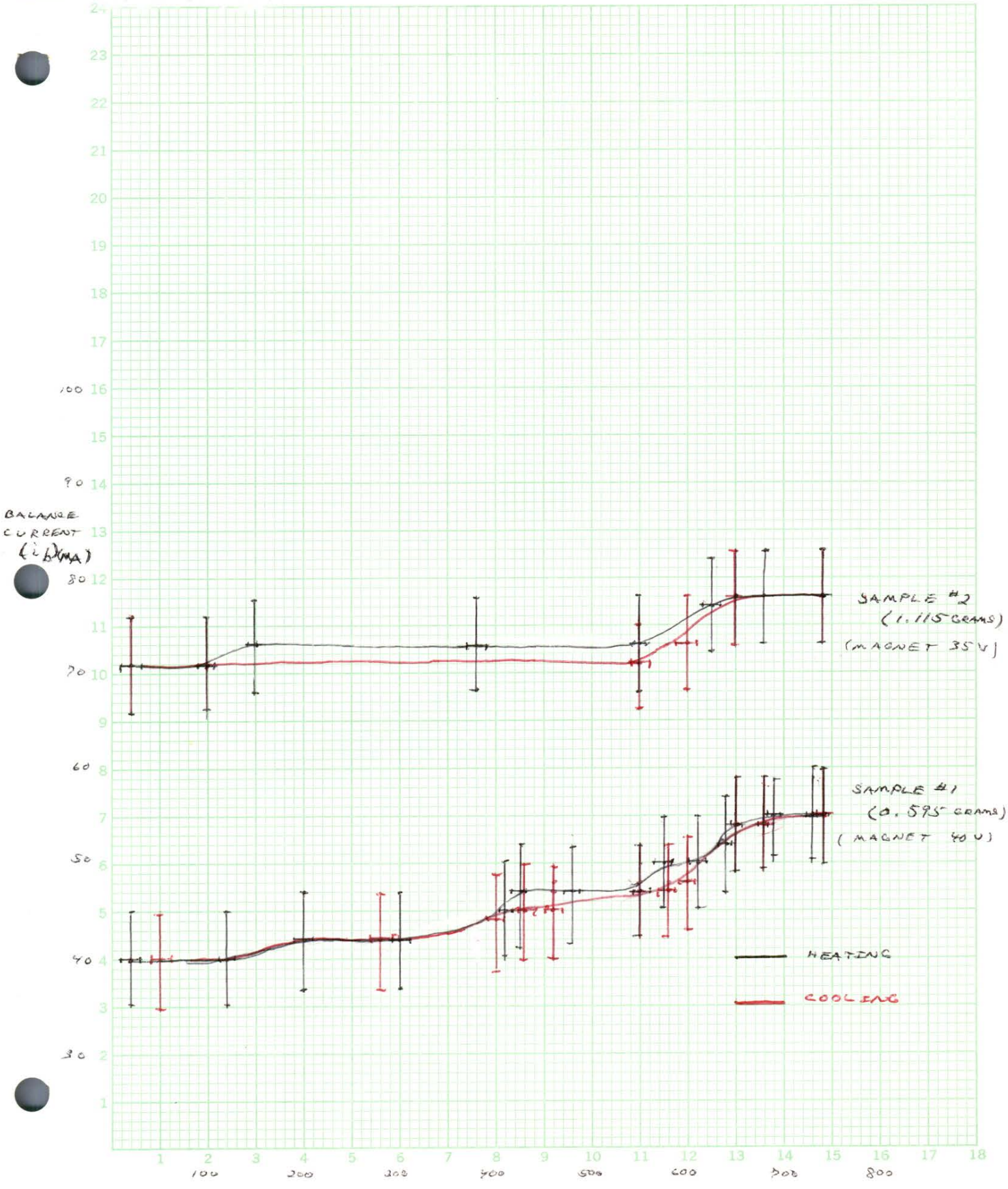
It is possible that this mineral is also an end member of an $FeTiO_3$ - Fe_2O_3 (ilmenite-hematite) solid solution series. This could explain the fact that the curie temperature is lower than those measured for natural hematite. That, the balance is incapable of measuring, for that is specifically dependent on chemical composition and can be determined by methods not presently found with the balance. Nevertheless, the proper magnetic reactions of ilmenite can best be studied at temperatures much lower than those possible with the present apparatus.

The error is again large (specifically, sample 2) for the deflection between the start and finish of the heating curve is quite small. Yet the specific points are consistent with sample 1

so as to be the actual points of interest.

ILMENITE #1, #2

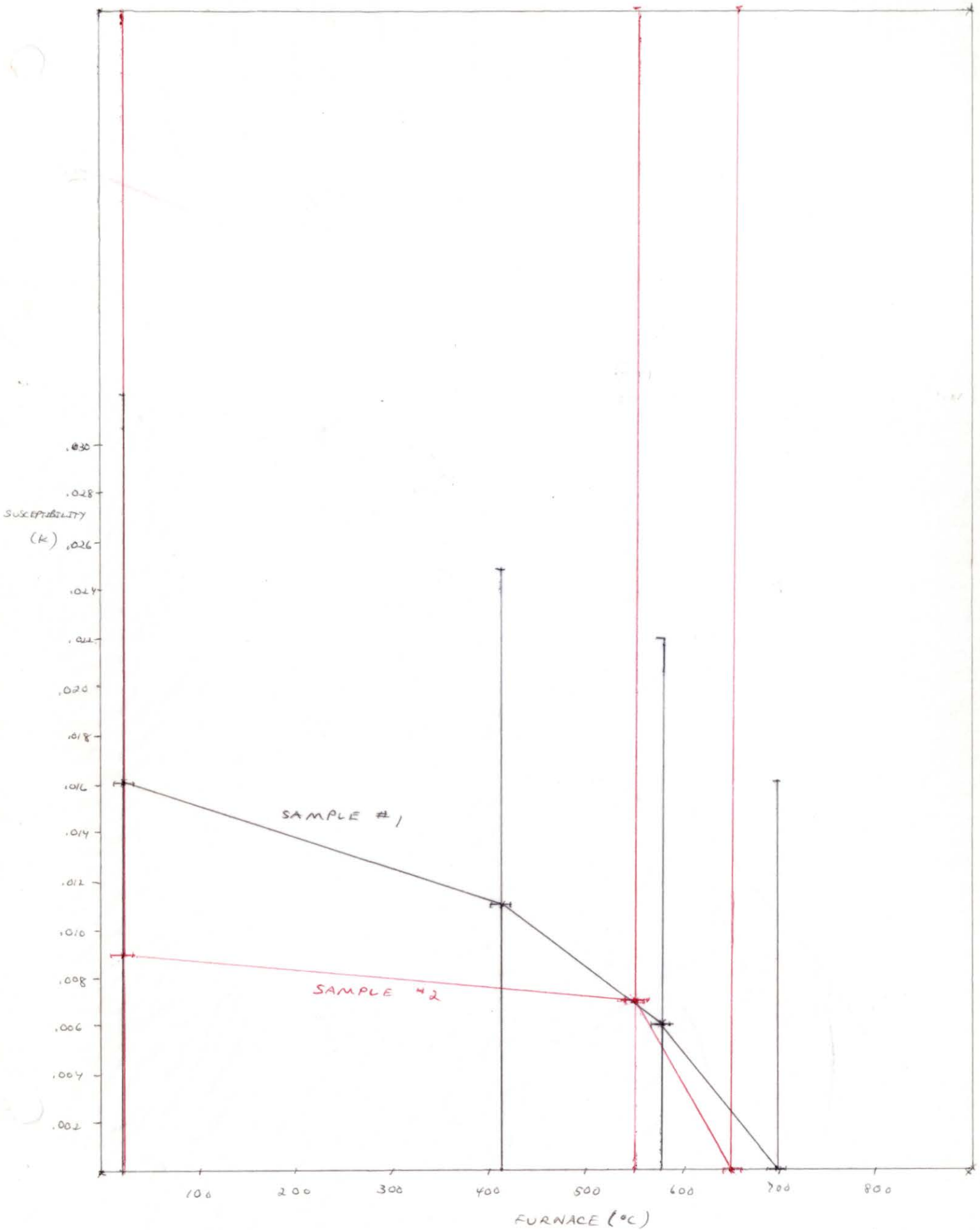
$FeTiO_3$



5 Squares to the Centimeter

ILMENITE

$FeTiO_3$



CONCLUSIONS

The thermomagnetic balance, despite the crudeness of its appearance, is actually a formidable instrument in paleomagnetic research. The accuracy with which it can measure not only Curie temperature and susceptibility, but also the hysterisis of any magnetic sample, is literally astounding. The process is truly tedious, but can be also quite rewarding.

No attempt was made to calculate the hysterisis of any of the minerals tested, although with more time it could have been accomplished. Since the gradient and the field strength had been measured, simple repositioning of the electromagnet poles can be utilized to increase the range of the field, and therefore the magnetization strength upon various rock samples.

No attempt was made to calculate an error for the Curie temperature, other than observational comments. It was felt that there are too many variables involved (i.e. chemical composition, time lag between furnace and sample, and the physical resistance to heat of some samples) to assess a proper error. Although it is directly related to the suceptibility, which was the chief objective of this paper, to assess an error in the calculations of susceptibility in reference to the Curie temperature, would be a blind action.

The basic difficulty with the balance is a proper assessment of error and ultimately the accuracy. As had been shown by the results of these tests, small susceptibilities had grossly large values for error. This is simply because the methods of observing and calculating these small values are crude. What does seem

amazing is that the actual values calculated show the general performance of the mineral as is theoretically estimated.

The one puzzing result that had been perpetrated through the entire experiment was the inability of the sample to reach a point of positive paramagnetism at the Curie temperature. What actually happened (as can be correlated from the Curie temperatures measured from the heating-cooling curves and the original balance calibration graph), was that the material went beyond the point of paramagnetism at the Curie point (the mass of the diamagnetic quartz of the cup is much greater than that of the sample, so overall, at the Curie temperature, the cup and the sample are theoretically paramagnetic). What this seems to indicate is that the effectiveness of the balance is so great that it defies electromagnetic laws, or else the position of the pendulum and the sample within it are not perfectly perpendicular to the field generated by the magnet. Naturally, the later is the most likely case, and it will be disussed later.

In summary, the experience was quite self-rewarding. One could ask what was the purpose of all the error calculations, since susceptibility is different for any rock or mineral measured, being directly dependent on a specific chemical composition. In answer to that it can be said that what it does provide is a range that the susceptibility is likely to fall. With that information, one can calculate a variety of data, from magnetization potentials to specific composition itself, with the calculated error always important. The results of this experiment show that the balance is an effective and relatively simple tool in paleomagnetic research, and its future use in this department is inevitable.

FUTURE IMPROVEMENTS OF THE APPARATUS

The most important step that can be made is to improve the accuracy of the balance fulcrum. (This is the sticking phenomenon mentioned earlier.) Although the razor blades are very accurate, a problem that occurred several times was that the upright supports that were glued onto the blades (and were blades themselves) continuously fractured. Repeated glueing caused the gap in which the copper crosspiece fitted to separate much too wide. The effect was that the balance would slightly shift and be somewhat off line from the direction of the elctromagnet field. This was repeatedly repaired, but it still lacked a complete solution. This resulted in a value of the balance current being larger than needed at the Curie temperature, since the distance to the center of the magnet had been increased. With any small displacement, this would be measured by the sensitive light source as an incalculable error. In the future, this will have to be accounted for prior to any testing.

The light source itself should also be improved. Although it was extremely sensitive, the focus of the spot on a calibrated graph sheet deflected off the mirror on the T of the pendulum was extremely crude. Preferrably, a translucent to transparent scale could be used as the recipient of a secondary deflection of the same light beam. This would more than quadruple the present sensitivity. The beam should also be focused (which was not done here) to prevent optical divergence. This can be done by mounting a lens near the mirror, but not attached to the balance mechanism.

Although it is a minor repair, if a 1000 milliamp-ammeter can replace the present 100 amp-ammeter on the magnet, a much more accurate balance will be available. As it was, in estimating the field and gradient from the information found at the rear of the paper from an estimated value of one amp (at 40 volts) and than extrapolated on those graphs downward was quite crude. Hopefully, this will be remedied before the next use of the balance.

The range of the minerals tested could be greatly improved by changing the atmosphere of the furnace. Perhaps, in some way, placing a vacuum pump over the furnace or having it contained within a non-reactive environment (i.e. helium-argon) would be an effective method. This would prevent early oxidation of certain minerals and therefore allow a greater range of observation.

Improving the atmosphere may also be accomplished by increasing the range of the minerals tested. This can be done by removing the furnace and replacing it with a suspended flask of liquid air. The engineering difficulties of this would be tremendous. How the experimenter would prevent the material from spilling over the balance mechanism presently seems impossible. However, if some other type of cooling mechanism were designed, such as a cooling coil, the range of minerals tested and their particular properties at those temperatures could be increased.

With improvements in the balance system and the light source a better assessment of error could be accomplished, much more so than in this experiment. It is the hope of this most recent investigator that such improvements can be made and implemented in the near future.

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Bibliography corrected with the assistance of the Senior Thesis advisor, Hallan C. Noltimier, on 7-18-14.

A FINAL NOTE

These last few graphs were measured during the summer of 1974 by Dr. Hallan Noltimier and Doyle Watts (a graduate student at the time). These allowed for the correct values of the electromagnet field strength and its gradient to be extrapolated. Since there was a hinderance as a result of having only a 100-amp ammeter instead of a 1000-milliamp ammeter on the magnet, this data was estimated using the following method:

Using the linear relationship of $V=IR$, and estimating 40 volts in the one amp position, the values of voltage along the scale of graph a was assessed. From graphs b, c, d, e, and f, the value of the gradient obtained from a was used to calculate the field. The graph used to calculate the field was selected by its nearest proximity of field current to what was estimated. (i.e. If the magnet was set at 15 volts, the value of the current corresponded to approximately 425 ma from graph a. Going to graph c, the proper value of the field could then be measured.)

NO. 340-M DIETZGEN GRAPH PAPER
MILLIMETER

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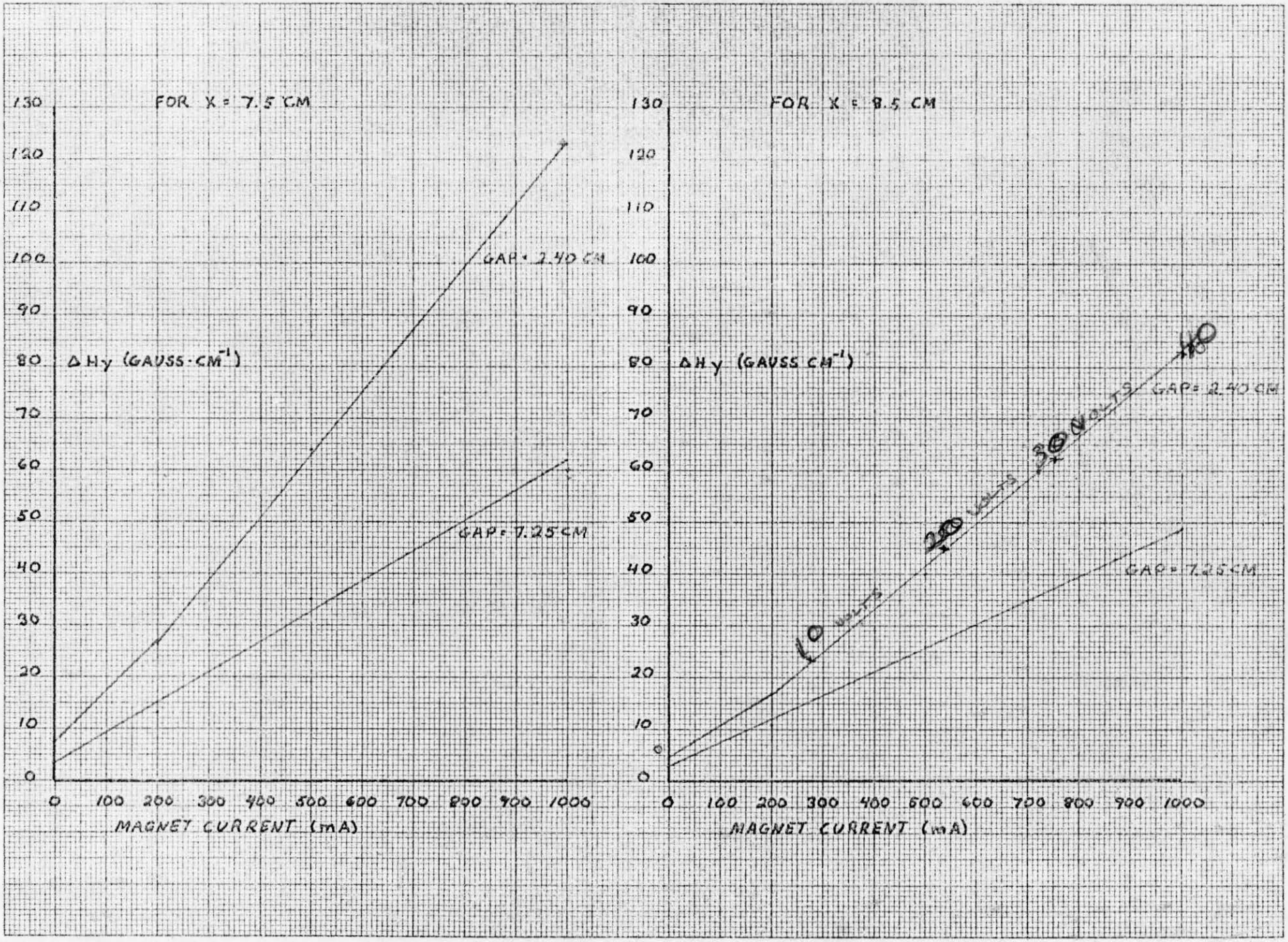
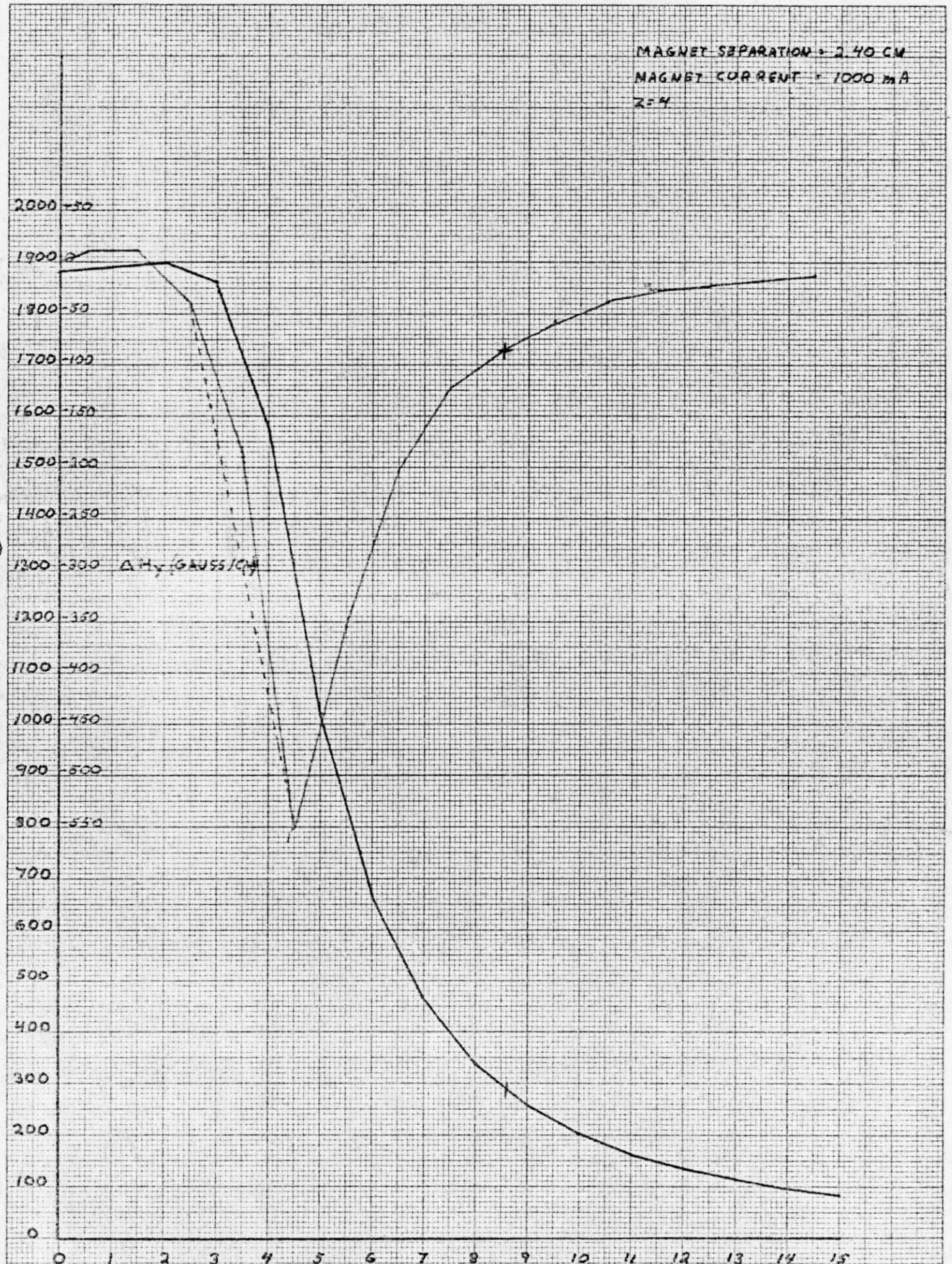
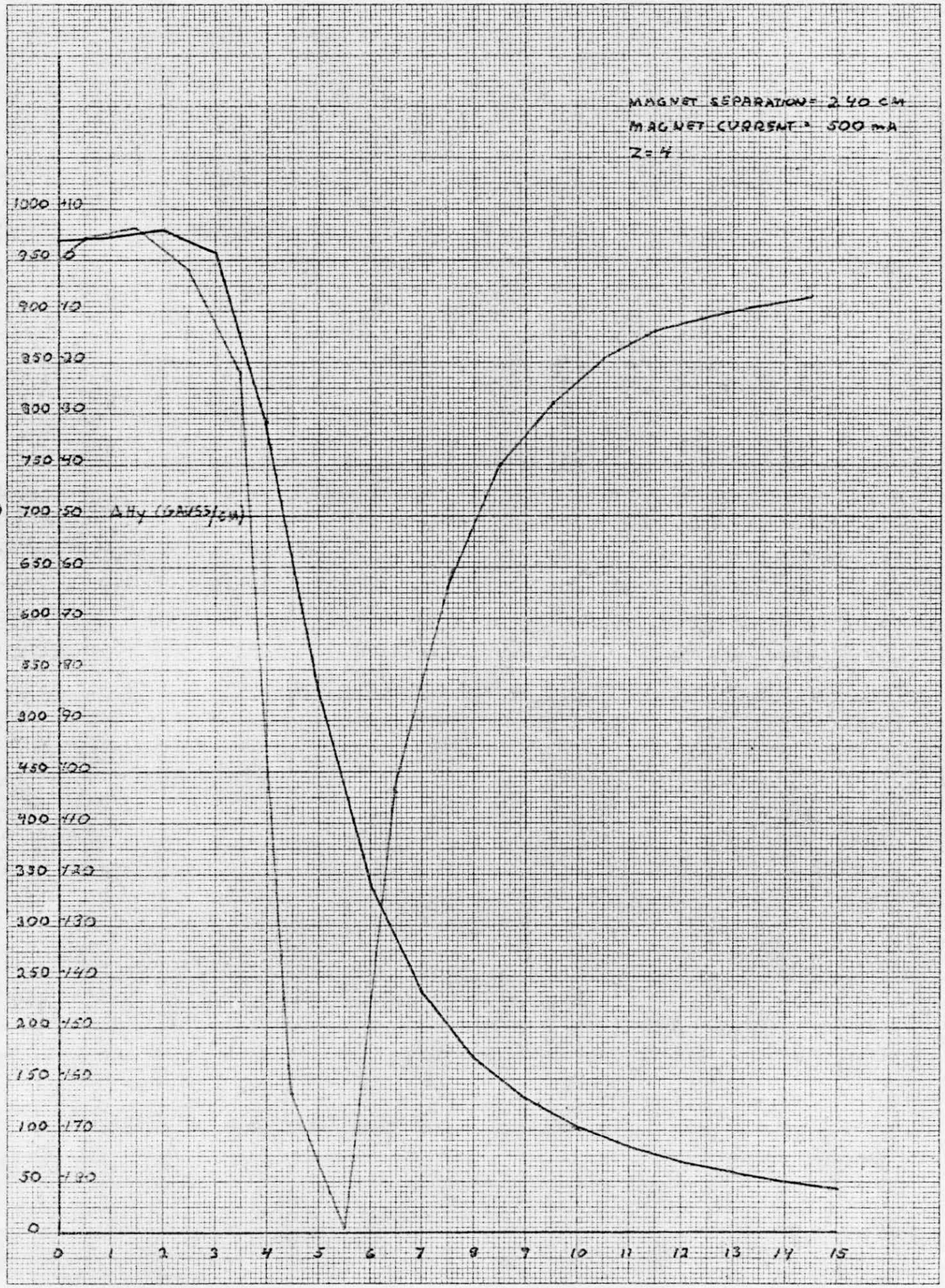


GRAPH A (a)

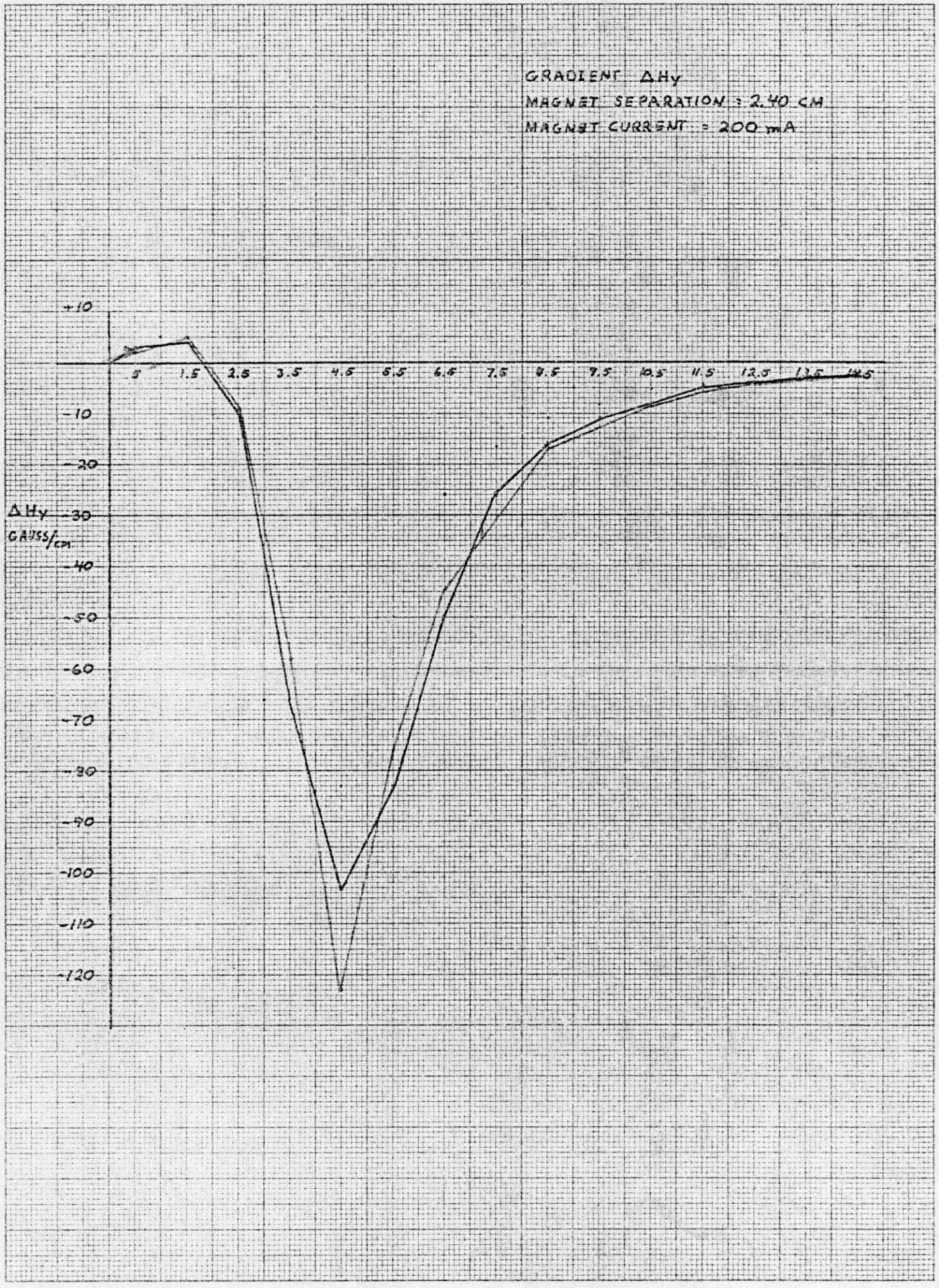
GRAPH B (6)



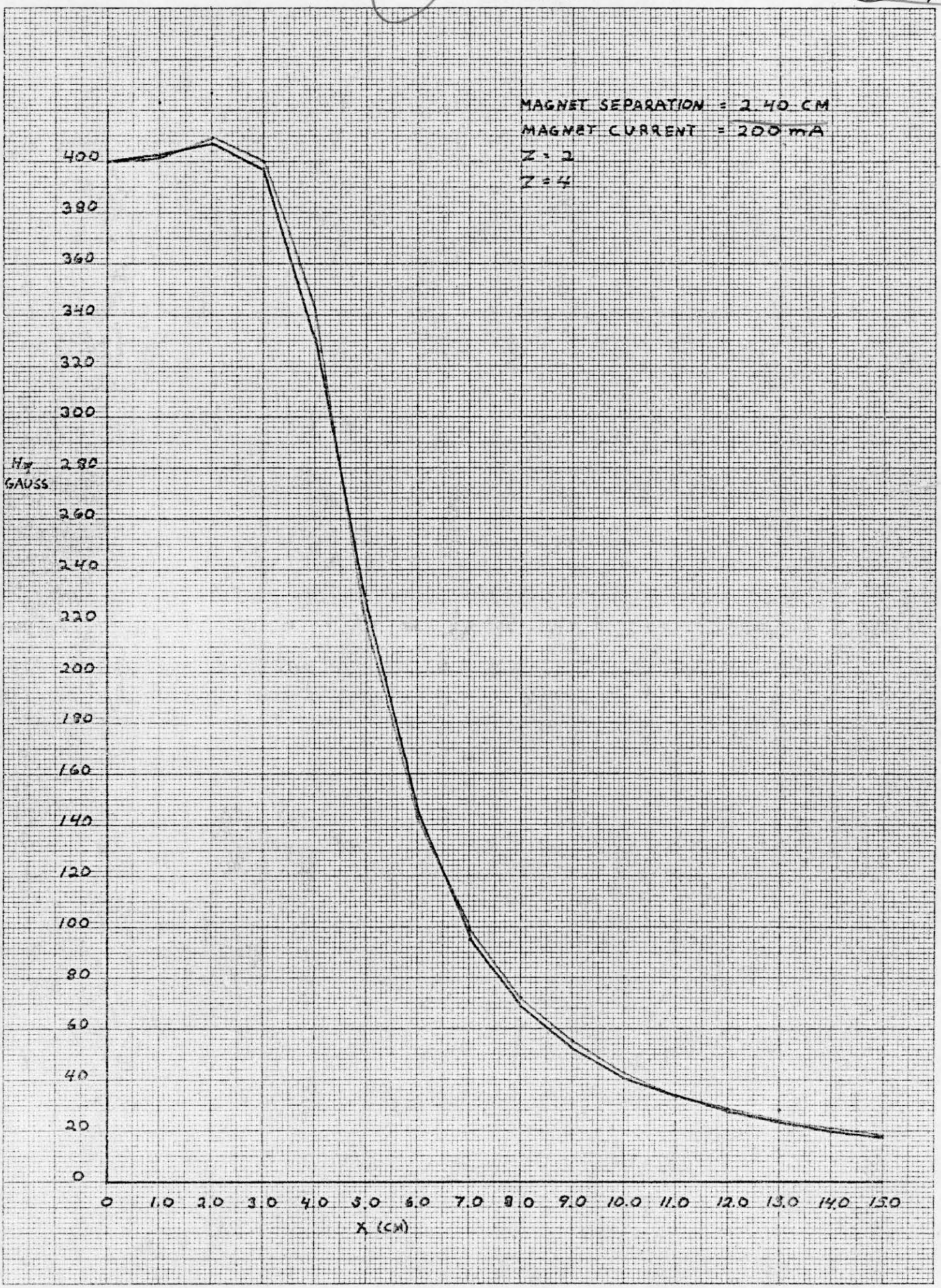
GRAPH C(c)



GRAPH D(d)



GRAPH
E (2



GRAPH F (8)